Wilcox Oil Company Superfund Site

Investigation of Lead Contamination at the Ethyl Blending and Lead Sweetening Areas

Bristow, Creek County, Oklahoma

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Summary

EPA investigated lead-contaminated soils in two areas of the Wilcox Oil Superfund Site in Bristow, Oklahoma. Soil samples from two intervals, 0 to 6-inches and 6 to 24-inches at the Lead Sweetening area (LSA) and Ethyl Blending Area (EBA) were analyzed for lead at the field office using an X-Ray Fluorescence (XRF) analyzer. The objective of the study was to delineate areas of lead exceeding the screening level of 200 milligrams per kilogram (mg/kg).

At the EBA, shallow (0 to 6-inches) lead contamination above the 200 mg/kg screening level was found throughout the sampled area (Figure 1.1) and likely extends beyond the area sampled to the south and east. Contamination in the 0 to 24-inch interval was identified on the north and south sides of the buildings. A definitive boundary of lead with a concentration of greater than 200 mg/kg in the 0 to 6 and 0 to 24-inch intervals was not identified. However, several areas were definitively identified as exceeding the screening level. These areas covered 10,227 square feet in the shallow and 3,660 square feet of soil in the deep DUs and represented 189 cubic yards of soil in the shallow and 68 cubic yards of soil in the deep DUs. Contamination from adjacent sources and former uses appears to overlap in the EBA study area.

Based on the preliminary LSA Conceptual Site Model (CSM) from the 2015 field investigation, the field team assumed that the highest concentrations occurred in the area's center. This CSM was used to guide the sampling approach and the team aimed to collect samples radially away from the LSA center until concentrations of less than 200 mg/kg could be confidently identified. Lead concentrations throughout most of the LSA exceeded the 200 mg/kg screening level and a definitive line delineating areas greater than 200 mg/kg could not be confidently identified. Real-time analysis of XRF data, field observations, and review of historic aerial photographs led to the revision of the working CSM during the field program. The revised CSM recognizes that high level of lead contamination from numerous identified sources exists throughout the Wilcox area and overlaps with the influence of the LSA source, obscuring the 200 mg/kg boundary. Lead particles have been transported by wind and vehicles on the main and secondary access roads. Areas with concentrations less than 200 mg/kg are relatively small and represent the exception rather than the base condition. The main access road and roads cut through the brush are contaminated and act as sources for further dispersal of contamination. The scale of heterogeneity of soils necessitates a decision-unit based incremental sampling approach for decision making.

The limited number of deep (0 to 24-inch) samples and distribution of samples with concentrations less than the screening level precluded geostatistical analysis to define a 200 mg/kg boundary, however the sample results define a minimum area of 1.9 acres above 200 mg/kg.

Results of the road and berm samples suggest that the main access road and the other built roads contain high lead concentrations that may act as sources if the road material is transported to other site areas. It is unknown if the original construction material contained lead or if the roads became contaminated as a result of transport from the LSA.

1. Introduction

1.1. Objective

The Technical Memorandum describes a focused investigation to identify the horizontal extent of lead contamination in the upper two feet of soil at the Lead Sweetening Area (LSA) and Ethyl Blending Area (EBA) at the Wilcox Oil Superfund Site. The objective of the study was to identify areas where lead concentrations in soil were greater than a screening level of 200 milligrams per kilogram (mg/kg). All work described in this Memorandum was conducted in accordance with the Quality Assurance Project Plan (QAPP) for The Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area at the Wilcox Oil Superfund Site (EPA, 2017), and the associated Standard Operating Procedures (SOPs). These documents present a detailed discussion of the problem statement, the conceptual site models (CSM), sample strategy, and quality assurance (QA) and quality control (QC) procedures used during project execution.

This Memorandum describes the elements in the approach that are different from what was proposed and presents the results and conclusions of the study and provides recommendations.

This Technical Memorandum is organized in four sections.

- **Section 1 - Introduction** describes the objectives of the investigation and the organization of the document.

- **Section 2 - Methods** is a brief summary of the methods used for assessing the sites and discusses deviations from the Work Plan and QAPP. This section includes the results quality control activities.

- **Section 3 - Results** describes the results of the sampling and analysis of the EBA and LSA areas and the Road Test Samples.

- **Section 4- Conclusions and Recommendations** presents the conclusions of the study as they relate to the objectives in the QAPP and provides recommendations for further actions.

2. Methods

The Interstate Technology and Regulatory Council (ITRC) has developed a guidance document that provides an extensive discussion on the theory and application of incremental sampling methodology (ITRC, 2013). Incremental composite sampling (ICS) methods specific to the Wilcox Soil Superfund Site are described in detail in the Work Plan (WP) for Investigation of Lead Contamination at the Ethyl Blending and Lead Sweetening Areas (EPA, 2017) and complete data QA/QC methods are described in QAPP for the Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area (EPA, 2017a). A summary of the sampling approach, work plan and QAPP deviations and QA/QC analysis for the LSA, EBA, and road test samples (RSTs) are described below.

The sampling design at the LSA was based on a CSM developed from preliminary site information and handheld XRF data from the 2015 SERAS investigation (Lockheed Martin, 2016). The sampling approach assumed, based on kriged and contoured data, the LSA contained a high concentration lead source in the center, and concentrations decreased away from the source. The general approach was to delineate a 200 mg/kg boundary for lead around the LSA source area and determine if the lead migrated vertically to the deeper soil (6 to 24-inch zone). The investigation aimed to evaluate the concentration of lead in the two potential exposure zones in upper two feet of soil (0 to 6-inches and 0 to 24-inches). The horizontal 200 mg/kg boundary was to be located using nine-increment sample unit (SU) composite samples along radial transects. Once the boundary was located, 30-increment decision units (DUs) were to be established to confirm the concentrations on the less than 200 mg/kg side of the boundary.

The sampling design at the EBA reflected an early CSM based on the limited knowledge and data regarding the history, source and existing conditions in this area from the 2015 EPA Emergency Response Team (ERT) investigation. The approach assumed that the building and its immediate proximity are the most likely source areas, and concentrations decrease away from the sources. The investigation aimed to evaluate the concentration of lead in the two potential exposure zones in upper two feet of soil (0 to 6-inches and 0 to 24-inches). The sampling strategy for determining if lead concentrations greater than 200 mg/kg were present (and if so, then delineating a 200 mg/kg boundary) began with a visual evaluation of existing conditions for indications of potential release mechanisms. The visual inspection for potential source areas (PSAs) included looking for material storage areas, product transfer points (such as piping or valves), release areas adjacent to doors, and any stained or distressed areas near the building. After PSAs were identified, DUs directly adjacent to the EBA buildings were developed and sampled. "Step-out" DUs were developed and sampled outward to determine if lead concentrations decreased with increased distance from the EBA buildings.

As a part of the adaptive CSM, the field team hypothesized that the site roads played a role in contaminant distribution and transport. Since roads were not considered in the preliminary CSM, a new sampling strategy was added to address sample collection on roads. Nine road sample tests (RSTs) were collected to help determine if road soils had high lead concentrations and if the road type was related to lead concentrations. Samples were collected from:
- "Built roads" - constructed with a gravel road base
- "Cut roads" - dirt roads made by cutting away brush and trees
- "Berm roads" – dirt roads on former storage tank retention berms

The field crew collected RSTs by first measuring a 100 ft section of road to be sampled and flagging the ends of each line. Ten shallow (0-3-inch) increments were collected at random locations evenly distributed across the 100 ft line (Figure 2-1). The field crew used small plastic trowels to collect the samples while avoiding gravel, debris and vegetation.

Figure 2-1: Schematic of RST configuration
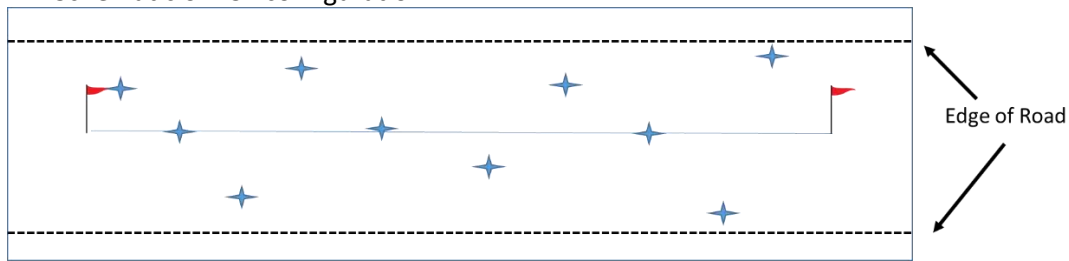
At the EBA and LSA, the field crew utilized in situ XRF core analysis, an adaptive sampling technique that was not described in the QAPP or WP. In situ core analysis was used to quickly indicate potential lead concentrations within the 24-inch core to help guide SU locations and overall decision making. After the deep soil core was extracted, it was laid down with the open window of the corer facing upward (Figure 2-2). XRF readings were collected in handheld mode every two inches along the exposed core and recorded on a sample collection sheet. To keep the XRF window clean and protected, a sheet of plastic (previously checked for XRF interference) was positioned between the soil core and the XRF instrument. The 22 to 24-inch reading was collected from the soil plug extracted from the soil corer tip.

Figure 2-2: In situ core analysis with the XRF in handheld mode.
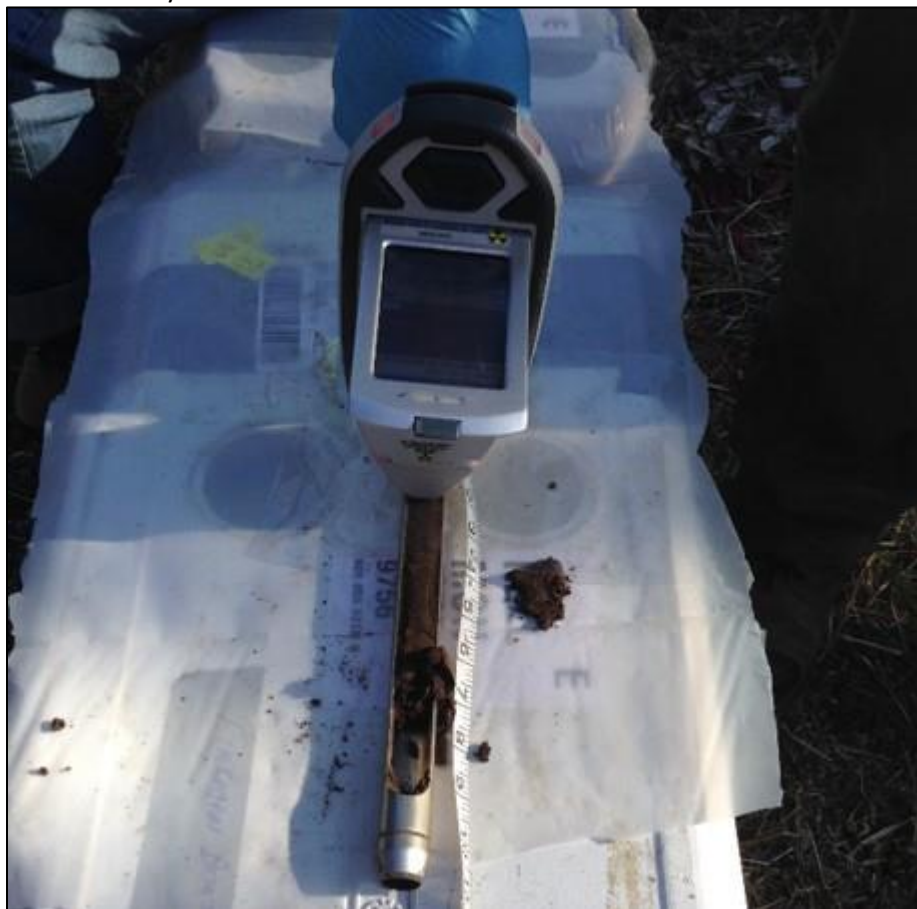
2.1.   Field and Laboratory Methods Deviations

Field methods followed the SOPs and the QC procedures in the QAPP, however, in some instances the field methods required modification or quality control procedures did not meet expected performance standards. This section describes the deviations and potential impacts on the data.

When the field team located the transect lines and initial points at the LSA, there were minor issues with the GPS navigation feature, so the live GPS screen was used to navigate to sampling points. As a result, some of the initial points and transect lines are not located in the originally planned location. All GPS issues were resolved before the final sample locations were recorded and reported less than one-meter precision for all locations. This does not impact the accuracy of the final sample locations.

Due to tree coverage and interference from the buildings, accurate GPS coordinates could not be recorded for many of the EBA sample locations. The field crew measured the boundaries of DUs off of the EBA buildings and recorded the measurements on a field data sheet. The DUs and EBA buildings were later digitized from an aerial image to create an EBA sample location map. The GPS had sufficient satellite coverage to record the location of the SUs north of the EBA buildings. Field measurement of the EBA DU boundaries does not substantially impact the accuracy of the final sample location figures.

At EBA DUs 3, 5, and 10, buried debris made it difficult to advance the soil corer 24-inches below ground surface (bgs) to obtain a full increment. For approximately five increments within each of these DUs, the corer was only advanced between 20 to 22-inches bgs, before the increment was incorporated into the composite sample. If probe refusal for any increment was encountered less than 20-inches bgs, the increment was recollected within the same DU and nearby the initial increment. Incomplete increments have negligible impacts on sample representativeness because very few of them were included in the final composite sample.

Site soils contained enough moisture and fines, so that core recovery was generally 100% at the EBA and LSA. Core compression, however, may have impacted sample representativeness when the field crew segregated the 0 to 6-inch and 6 to 24-inch interval of a full soil core. To ensure a consistent and efficient workflow, the crews estimated the total compression of a core and used the estimate to determine the actual location of the 0 to 6-inch interval. If there was error in the compression estimate, portions of the 0 to 6-inch interval could have been collected with the 6 to 24-inch interval, and vice-versa. Since only a few increments from each DU were affected, there are negligible impacts on sample representativeness.

While collecting in situ XRF readings on soil cores, the field crew noticed that the interval at the very bottom of the open core window showed unexpected high lead readings. Upon further examination, it was clear that as the corer was extracted, the bottom portion of the open core window, approximately 22-inches down the probe, was scraping and collecting soil from the top six inches of the soil column (Figure 2-3). This scraping could impact sample representativeness because shallow soil was incorporated in the deep soil sample. The rest of the field crew was instructed to remove the excess soil from the core before incorporating the increment into the composite sample. This scraping and collection of soil was unlikely to impact sample representativeness because it was mitigated by the second day of field sampling.

Figure 2-3: Photo of scraped shallow soil collected at the bottom of the core window

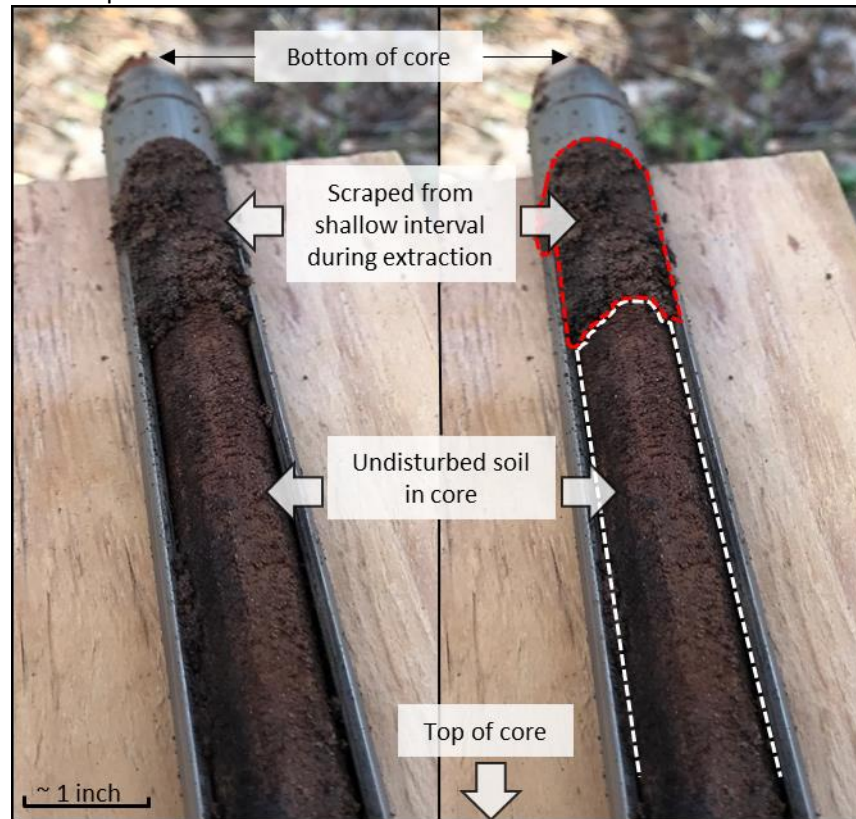Table 2-1 describes specific performance measures from the WP and QAPP that were not met or were added. The majority of deviations from the WP and QAPP were caused by physical restrictions in the field that limited sample collection as described in the WP and QAPP. Deviations were also initiated by changes in the lab and in the adaptive CSM that field personnel made as samples were processed and analyzed.

Table 2-1: WP or QAPP deviations and additions due to physical constraints, changes in the CSM, or changes in the lab

Grouping	Description	Implication
CSM	Addition of in-field XRF core analysis and bagged interval core samples	• Helped build CSM in the field by providing high-resolution information on how lead concentration changed with depth • Saved sample collection time
CSM	Road Test samples – driven by adaptive CSM	• Helped build CSM by showing that site roads may have contributed to contaminant transport • Preliminary info on lead levels on different road types
Physical	Bedrock encounter at LSA	• Prohibited deep sampling in the LSA SUT 16 area • Shows that contamination in that area is restricted to the surface
Physical	Railroad siding access restriction at EBA	• Three samples were collected between the NW side of the EBA building and the fence separating the site and the railroad right of way • No samples were collected beyond the fence
Physical	Inaccessible areas at ravine edges of creeks	• Steep terrain prevented access to the areas south, southeast of LSA transects 7, 8 and 9 • Uncertainty of contamination south/southeast of ravine
Physical	Modified core volume reduction method for DU samples to use a split core technique instead of the "window cut" approach in the QAPP	• QAPP required field crew to reduce sample volume from the 6-24-inch samples by scraping a flat edge across the open "window" of the corer, removing approximately one-third of the sample • Because the "windows" of the corers used in the field only exposed approximately ¾ of the sample, a split core technique was used • Revised technique increases sample volume and decreases sample bias
Physical	Sample disposal	• Specific sample disposal procedure (including location) after analysis was not expressly written into the WP or QAPP • With approval from the RPM, samples were removed from the sample bags and deposited in the area near LSA SUT 14,2.5. (Area within the LSA with high lead levels)
Lab	Olympus XRF not used	• Only the Niton XRF was used to analyze soil • Additional XRF was not used due to high demand for sample collection and processing • Did not impact data quality
Lab	100-mesh sieves not used	• 100-mesh sieves were not available on site • 80 and 120-mesh sieves used instead • Meshed fractions did not impact decision making at EBA or LSA
Lab	System check not conducted some days	• No Check (11/2/17 PM through 11/4/17) • No issue with the LCS QC during this period • Negligible if any effect on accuracy

3. Results

3.1. EBA Results

At the EBA, the field crew collected ten shallow SU, two shallow PSA samples, five deep SU and two deep PSA samples. 17 DU samples were collected at the 0-6-inch interval and six DUs were sampled at the 6-24-inch interval. Table 3-1 shows the total number of samples analyzed for the EBA. Full sampling results are available in Appendix 1.

Soil samples at the EBA were generally silty sand with some gravel. Buried debris was encountered within the majority of the EBA DUs as indicated by probe refusal. The area southeast of the EBA buildings, within DU 5 and some of DU 10, had buried black asphalt-like debris that ranged from 0 to 24-inches bgs. This debris made it difficult for field crews to drive and extract deep soil cores in some areas. Black debris and a dark, oily product with a strong petroleum odor were encountered north of the EBA buildings in the vicinity of DU 3 and EBA SUT 1,1.

Table 3-1: EBA Sample result summary

Sample Type	Depth	Number*	Samples >200 mg/kg
Sampling Unit†	0 to 6-inches	12	9
	6 to 24-inches	7	1
Decision Unit‡	0 to 6-inches	23	12
	6 to 24-inches	6	1
Ramp Transect†	0 to 6-inches	2	2

*Includes triplicate samples
†For SUs and Ramp Transects the mean concentration of the bagged sample was used for decision making.
‡For DUs the 95% UCL of the bagged sample was used for decision making.

The EBA investigation started with sampling at PSAs near the southeast building (Figure 3-1). PSA 1 included the valves and pipes adjacent to the southeast side of the southeast building, and PSA 2 included a conical structure south of the southeast building that may have supported a storage tank. Lead in the shallow samples from both PSAs was greater than 200 mg/kg. Lead in the deep PSA 1 sample was greater than 200 mg/kg and the investigation was expanded using in-situ XRF readings of core samples collected up to 20 feet away from the pipes. Results of the core analysis are shown in Figure 3-2. Figure 3-1 shows the approximate extent of lead greater than 200 mg/kg in the 6-24-inch interval at PSA 1. Lead concentration was less than 200 mg/kg in the deep PSA 2 sample. PSA 1 was located within DU 1, and PSA 2 was located within DU 6. DU increments were not collected within the PSA areas. (See photos in Appendix 2)

Figure 3-1: EBA PSA 1 location and results
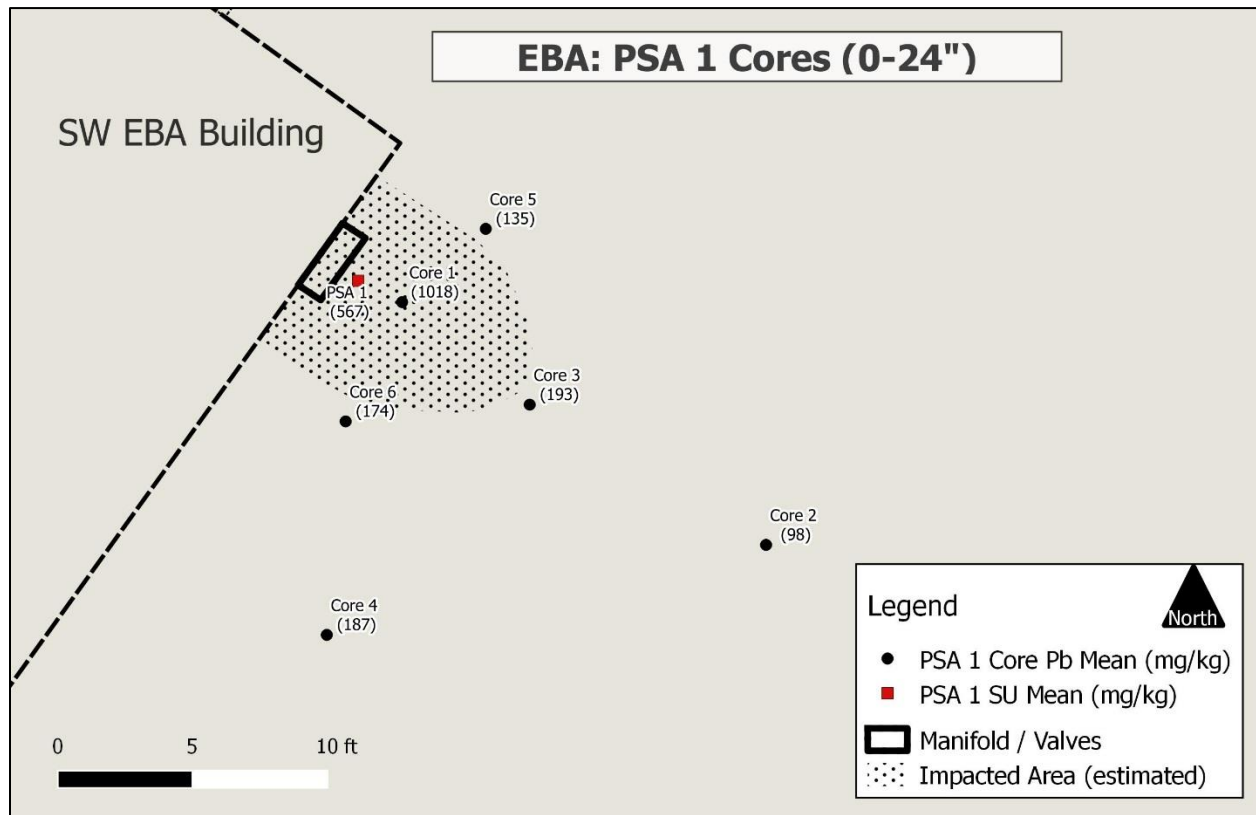
Figure 3-2: EBA PSA core analysis results
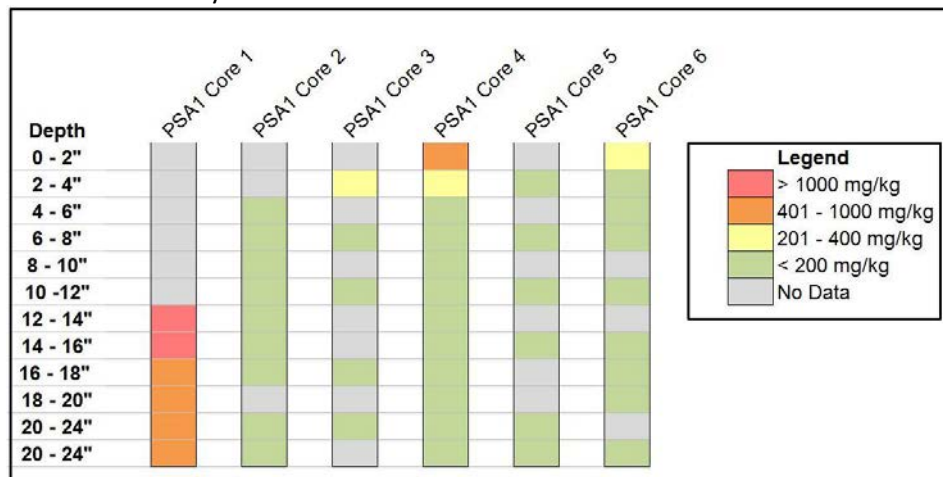
After the PSAs were sampled, the field team sampled shallow DUs adjacent to the EBA buildings. DUs that had lead concentrations greater than 200 mg/kg were DU 5 to the southeast, DU 6 to the south, DU 1 to the southwest, DU 3 to the northwest, DU 4 to the north, and DU 8 between the two buildings (Figure 3-3). DU 9, located within the southwestern EBA building, was also above 200 mg/kg. Shallow DUs adjacent to the EBA buildings with lead concentrations less than 200 mg/kg included DU 4 to the north and DU 2 to the northwest. Lead levels in shallow transects on the earthen "ramps" leading into the southeastern building were also above 200 mg/kg. A concrete containment structure filled with rainwater was identified on the south side of the EBA. Although it was not identified as a PSA, sludge

was sampled from the bottom of the containment structure and contained less than 200 mg/kg lead. Five SUs were collected along a transect at the edge of the road north of the EBA to get an understanding of the concentration trends to the north of the EBA building. All five SUs showed concentrations of lead greater than 200 mg/kg in the 0 to 6-inch interval.

After initial DUs adjacent to the buildings were sampled, shallow "step-out" DUs were collected to evaluate if lead concentrations decreased away from the EBA buildings. The goal was to delineate the boundary of lead contamination at the EBA with DUs less than 200 mg/kg lead by collecting step-out DUs. DU 10 (step-out from DU 5) and DU 16 (step-out from DU 10) were collected southeast of the EBA buildings. DU 7 (step-out from DU 6), DU 14 (step-out from DU 7), and DU 17 (step-out from DU 14) were collected southeast of the EBA buildings (Figure 3-3). Although DU 12 was below 200 mg/kg lead, DU 15 was collected to evaluate lead concentrations north of the EBA adjacent to the SU transect, where concentrations exceeded 200 mg/kg and process structures were identified. DU 15 showed lead concentrations less than 200 mg/kg. Additional step-out DUs were not collected outward from DU 16 and DU 17 because these step-outs would be located in the vicinity of adjacent process areas, as identified by Sanborn maps, that are not associated with the EBA.

To delineate the depth component of lead contamination, six deep DUs and four deep SUs were also sampled at the EBA. Lead concentrations for the 0 to 24-inch interval were calculated using a weighted average at sample locations where both 0 to 6 and 6 to 24-inch intervals were collected (Figure 3-4). Lead in DU 3 and DU 10 was greater than 200 mg/kg, and lead concentrations in DU 1, DU2, DU 4 and DU 6 were less than 200 mg/kg. Lead concentrations in the deep SU 1 and PSA 2 samples was less than 200 mg/kg, and all other deep SU samples were greater than 200 mg/kg.

Figure 3-3: EBA 0 to 6-inch sample locations and results

Figure 3-4: EBA 0 to 24-inch sample locations and results

3.2.   LSA Results

At the LSA, 174 samples covering approximately 10 acres were collected, processed and analyzed. The field crew collected 84 shallow and 13 deep SU samples, ten shallow DU samples, 9 RSTs, and ten deep core samples. Table 3-2 shows the total number of samples analyzed for the EBA. Full sampling results are available in Appendix 1.

Table 3-2: LSA Sample collection summary

Sample Type	Depth	Number*	Samples >200 mg/kg
Sampling Unit†	0 to 6-inches	84	48
	6 to 24-inches	12	11
Decision Unit‡	0 to 6-inches	8	4
Road Tests†	0 to 6-inches	9	4
Cores†	4-in increments 0-24-in	60	44

*Includes triplicates and couplets

†For SUs, Road Tests and Cores the mean concentration of the bagged sample was used for decision making.

‡For DUs the 95% UCL of the bagged sample was used for decision making.

Soil samples at the LSA were typically brown fine sand or silty sand. Subsurface debris, indicated by probe refusal, was encountered at some LSA locations, but not as frequently as the EBA. Bedrock is exposed on the main road between LSA SUT 16,1 and 16,2 and a deep sample near LSA SUT 16,1 could not be collected due to shallow bedrock less than one-foot bgs. LSA sample collection was also restricted by a ravine that runs south from the retention pond berm east of LSA SUT 7,3 and 8,1 and cuts along the south of the LSA by LSA SUT 9,1 and 8,2. No samples were collected on the far east or far south side of the ravine.

The LSA investigation started with collecting SU samples where each of the 16 radial transects intersected the kriged 200 mg/kg boundary from the ERT investigation. These preliminary samples are labeled LSA SUT X,1 on Figure 3-5. If the lead concentration of the preliminary SU was less than 200 mg/kg, a "step-in" sample was collected towards the center of the LSA. If a preliminary SU was greater than 200 mg/kg, a "step-out" sample was collected away from the LSA center. DUs were sampled between adjacent transects when a set of confirmatory couplet SUs showed lead concentrations less than 200 mg/kg (Figure 3-5). The only DU with a lead UCL less than 200 mg/kg was LSA DUT 4-5. The remaining three LSA DUs, LSA DUT 5-6, 6-7, and 8-9 had UCLs above 200 mg/kg.

DU samples were not collected in the north/northeast boundary of the LSA because SU samples at the furthest point were considerably greater than 200 mg/kg lead. Mean lead in a couplet at LSA SUT 2-3,3 northeast of the LSA was 1726 and 1319 mg/kg and LSA SUT 1,4, the farthest north sample, was 3119 mg/kg. DUs were not developed west of the LSA because mean lead in SUs at the most distant locations (LSA SUT 15,4, 15,3 and 13,5) were above 200 m/kg. Although several SUs southeast of the LSA showed mean lead concentrations less than 200 mg/kg, mean lead results from the LSA SUT 11,3 couplet further out were 1017 and 43575 mg/kg. The highest lead concentrations were found in the center of the LSA at LSA SUFHI 1 and 2 with means of 11,825 and 170,142 mg/kg respectively. (See photos in Appendix 2).

Lead concentrations for the 0 to 24-inch interval at the LSA were calculated using a weighted average at sample locations where both 0 to 6 and 6 to 24-inch intervals were collected (Figure 3-6). The highest 0 to 24-inch lead concentrations were sampled in the center of the LSA at LSA SUFHI 1 and 2 with means

of 11,513 and 55,858 mg/kg respectively. The field crew attempted to collect a deep sample along transect 16. However, bedrock was encountered between 8 to 12-inches bgs around LSA SUT 16-1 and further downgradient of transect 16. The only deep sample with lead concentrations less than 200 mg/kg was LSA SUT 2,1 northeast of the LSA.

Eight 0 to 24-inch core samples were collected at the LSA and analyzed in 6-inch increments to help determine the distribution of lead with depth. Five of the samples were positioned near the inner ends of the transects and three core samples were collected in the vicinity of LSA SUT 9,2 south of the LSA to evaluate the possibility of a hot spot at LSA SUT 9, 2 (Figure 3-6). Figure 3-7 shows the concentration profiles of the cores with depth. The average lead concentration for all core samples was greater than 200 mg/kg, but the distribution of the lead in the cores varied considerably. Near LSA SUT 9, 2 the average lead concentration in the samples was 2692 mg/kg in LSA SUT 9,2N, 312 mg/kg in LSA SUT 9,2S, and 1208 mg/kg in LSA SUT 9,2W.

Figure 3-5: LSA 0 to 6-inch sample locations and results

Figure 3-6: LSA 0 to 24-inch sample locations and results

Figure 3-7: LSA core analysis results

Data from the deep cores and shallow sample locations along selected transects were used to prepare three cross sections at the LSA shown in Figure 3-8. Cross section A-A' cuts through the LSA from the northwest to southeast and shows the deep contamination in the center of the LSA and lower concentrations (below the screening level) near the outside edges of the transects. Cross section B-B' runs north-south through the LSA and also shows the high contamination at depth near the center of the LSA but lower concentrations in the deep sample from just north of the center at LSA SUT 16,3. The northernmost sample on cross section B-B' appears to show effects of the overlapping lead source from the product tank area to the north. Cross section C-C' cuts through the southeast part of the LSA and illustrates a broad area of contamination above 200 ppm and the edges where the extent was not identified.

Figure 3-8: Cross-sections A-A', B-B', and C-C' for 0 to 6-inch, 6 to 24-inch, and core segment samples at the LSA
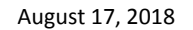
3.3.   Road Sample Tests

The field team collected RSTs as a part of the adaptive CSM to determine lead concentrations on the different types of site roads (Figure 3-9). The need for measuring the lead concentration of road surfaces developed during analysis of SU sample results near roads. The team observed that samples near the roads often exhibited higher concentrations than expected and SU couplets near roads had poor agreement. Samples collected on tank berms, which were also used for site access, appeared to have much lower concentrations than nearby samples. The team hypothesized that contaminated roads may influence sample results if SUs were located near roads or in areas affected by road sediment runoff. Two different types of roads were observed onsite. Built roads, providing access to the main processing areas of the Wilcox Refinery, consisted of boulder and gravel size rock with densely packed fine-grained soil between the rocks. These roads were commonly elevated above the surrounding area. Cut roads were generally built of soft soil and appeared to be more recently cut through trees to provide access to various areas of the site.

RSTs 1-3 and 5-7, collected on built and cut roads east and southeast of the LSA, showed mean lead concentrations greater than 200 mg/kg. Results from RST 8 and 9, collected on storage tank retention berms southeast of the LSA, were both less than 200 mg/kg. RST 4 collected on a cut road showed a mean lead concentration of 195 mg/kg.

Figure 3-9: RST locations and results
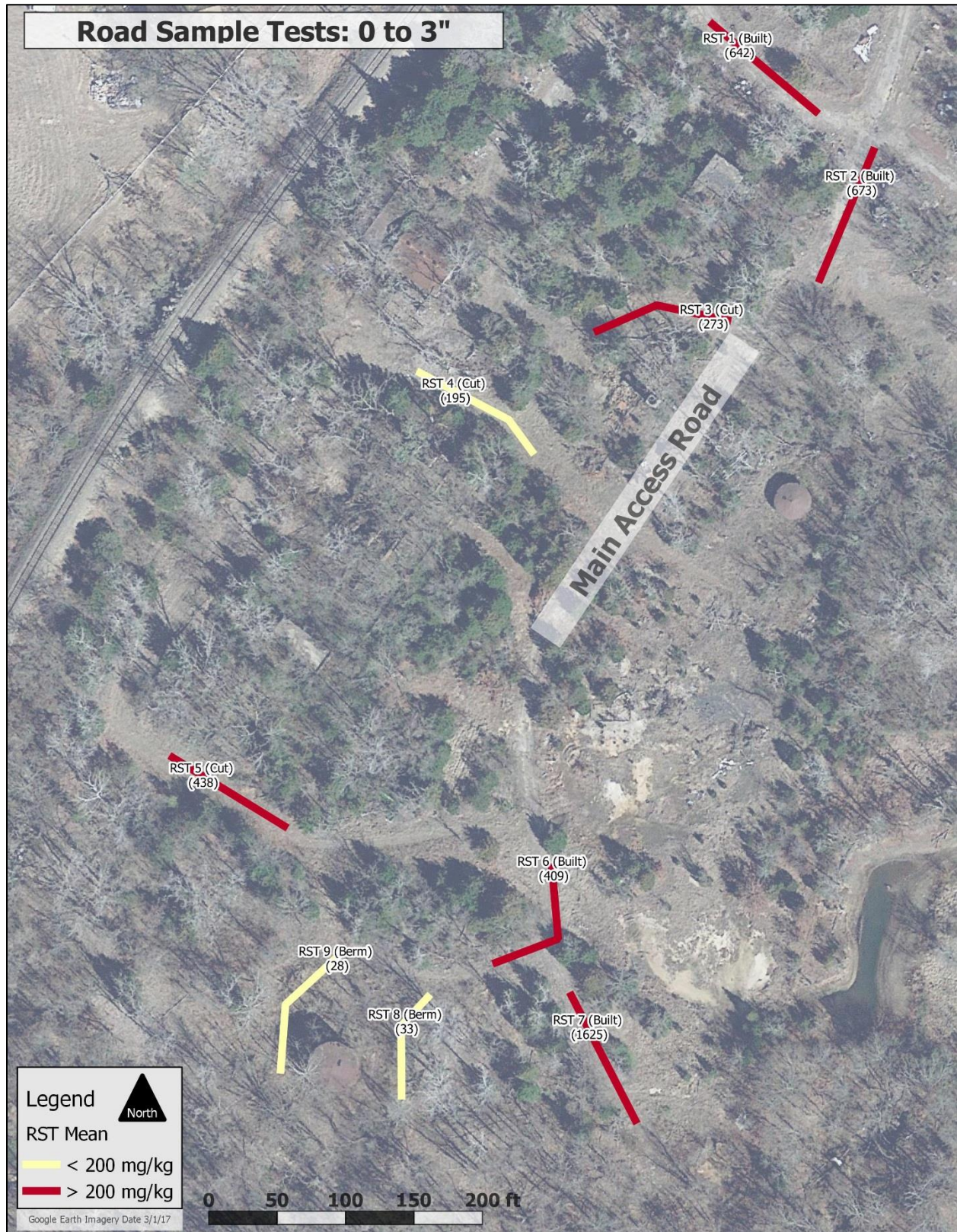
3.4.   Wilcox Oil Soil Particle Size Evaluation

This evaluation examined whether soil particle size influences lead concentration to address the questions about potential enrichment of smaller particle sizes for risk assessment, and the design of any potential future lead-in-soil sampling in this area of the Wilcox Site.

It is frequently found that metal concentrations in smaller soil fractions are significantly higher than the concentration of fractions that include larger particle sizes. This is important for risk assessment since smaller particle fractions are more likely to adhere to children's skin to complete the ingestion pathway. This pathway is important both when a child is exposed to outdoors soil and to inside dust derived from that soil. For that reason, EPA Superfund guidance recommends sieving soil to obtain the <100-mesh (i.e., less than 150 micron) particle fraction for lead analysis (EPA, 2016).

Since additional sieving beyond 10-mesh adds to the sample processing workload, it is useful to know whether there is a significant difference between the concentrations of the <10- and <100-mesh fractions. If the concentrations are not significantly different for a particular site, some cost savings could be realized without endangering the protectiveness of comparisons with screening level values.

The work plan called for testing particle size concentration differences with a 100-mesh (<150 microns) sieve, but that size sieve was unavailable for site work. Therefore, 80- and 120-mesh sieves were used to bracket the 100-mesh size fraction. Three samples were sieved with 1) a 10-mesh sieve to produce a soil fraction containing all particles smaller than 2 mm in diameter; 2) a 80-mesh to produce a soil fraction containing all particles smaller than 175 microns in diameter; and 3) a 120-mesh sieve to produce a soil fraction containing all particles smaller than 125 microns.

The sample IDs were EBA DU-10B (6-24-inch depth interval), EBA DU-5 (0-6-inch depth interval), and LSA SUT 5-6B (0-6-inch depth interval) (Table 3-3). Statistical evaluation used Analysis of Variance (ANOVA) to test for statistically significant differences among the three fractions at the 95% confidence level. If the ANOVA found significant difference (i.e., p-value <0.05), follow-up multiple comparison testing was done with Tukey's method to determine which fractions were different from each other.

Table 3-3: Mean lead concentration for samples sieved to multiple fractions

Sample ID	Depth	Mean Concentration <10 mesh (mg/kg)	Mean Concentration <80 mesh (mg/kg)	Mean Concentration <120 mesh (mg/kg)
EBA DU-10B	6 to 24-inches	122	99.8	130
EBA DU-5	0 to 6-inches	569	708	874
LSA SUT 5-6B	0 to 6-inches	158	148	189

- *Sample ID EBA DU-10B (6-24):* The lead concentration of the <120 fraction concentration was higher than both the <80- and <10 fractions, by 30% and 7%, respectively. These differences were not strictly statistically significant by analysis of variance (ANOVA), however significance was almost attained since the ANOVA p-value was 0.064.

- *Sample ID EBA DU-5 (0-6):* The lead concentration of the <120 fraction concentration was statistically greater than both the <80- and <10 fractions, by 23% and 54%, respectively. The ANOVA p-value was 0.0000.

- *Sample ID LSA DU 5-6B (0-6):* The <120-mesh fraction concentration was statistically greater than both the <80- and <10-mesh fractions, by 27% and 19%, respectively. The ANOVA p-value was 0.0000.

Conclusions from particle fractionation study can be summarized as below:
- Although the <100-mesh fraction could not be compared directly to the <10-mesh fraction, these results indicate that there would likely be a modest enrichment of lead in the smaller particle fraction.
- The degree of enrichment appears to vary with concentration.
    - At concentrations less than 150 ppm, the degree of enrichment appears negligible.
    - At concentrations closer to 200 ppm, it appears that mild enrichment could be possible.
    - At higher concentrations, lead enrichment could be as much as 50% higher in the <100-mesh fraction compared to the <10-mesh fraction.

3.5. Quality Assurance and Quality Control Results

Both incremental sampling and QC-assured XRF analysis are relatively new strategies for Superfund sites. Therefore it was important that the Wilcox Oil project demonstrate quality assurance (QA) practices that document the performance of these techniques. Details of the QC procedures used for the Wilcox Oil Lead Characterization project are covered in the Wilcox Oil QAPP. Results of the QC procedures are discussed in this section. Full QA/QC results are available in Appendix 3

3.5.1. Sample Collection QA/QC

Two types of soil sample units were defined for this site: SUs and DUs. SUs were small-area (2 x 2 ft) 9-point composite units used to characterize lead concentration trends to determine the placement and configuration of DUs. An SU sample bag mean (i.e., the arithmetic mean of XRF replicate data on a sample bag) is used as the "sample result" when further statistical analysis of the site SU data as a whole is done. For the Wilcox Oil project, further statistical analysis involved geostatistical modeling to provide lead iso-concentration contours. Contour uncertainty is reported as part of the modeling process. An SU sample bag UCL on the mean is used as the "sample result" to accommodate analytical uncertainty when a decision is made by comparing a single SU sample result to an action level. For the Wilcox project, this decision applied to deciding whether additional SU placement was required to locate the putative 200 mg/kg hotspot boundary.

QC checks in the form of SU "couplets" were used to assess the ability of an SU to represent the lead concentration for its surrounding area, and to confirm indications that a segment of 200-mg/kg boundary was found. This was accomplished by placing an identical SU approximately 2 ft. from the original SU along the putative boundary line. This was especially important at the start of the project to make sure that the initial SU configuration could provide representative data. A spreadsheet calculator was used to rapidly assess the agreement between the sample bag results (as bag means) of the couplets (shown below). Agreement was considered acceptable if the lower member of the couplet was within 30% of the higher couplet: (higher – lower)/higher ≤ 0.3 The 30% goal was specified in the QAPP after consideration of how much disagreement could be tolerated around the 200-mg/kg threshold before triggering an unacceptable decision error (a range of 140 – 286 mg/kg). The most unacceptable error was deciding that the 200-mg/kg boundary was reached when in fact it was not. This meant that the narrower interval needed to be on the lower side of 200 mg/kg. Even occasional disagreements in SU couplets near the 200-mg/kg threshold would have been reason to alter the SU configuration (i.e., enlarge the SU area and increase the number of increments) to produce more representative samples.

A total of 20 SU couplets were sampled. Six of the 20 couplets exceeded the 30% goal; and only one of those occurred with one of the couplet results within the 30% band around the 200-mg/kg threshold. These disagreements indicated the degree of short-scale field heterogeneity present at this site, especially at higher concentrations. In particular, we attribute some of this variability to the influence of roads at certain sample locations, and the possible influence of soil scraping at LSA Test1-SUT1 (6-24). However, the frequency of overall agreement (70%), and agreement around 200 mg/kg (95%) was considered sufficient to continue using the initial 2 x 2, 9-pt SU configuration (Figure 3-10)

Figure 3-10: SU couplet evaluator results

SU Couplet Evaluator					
SU Identifier	Highest couplet conc	Lowest couplet conc	minus 30% of highest	Is the lowest within 30%?	Actual % diff (rel to highest)
LSA Test1-SU1 (0-6)	3279	3141	2295.3	yes	4.2
LSA Test1-SU1 (6-24)	880	296	616	no	66.4
LSA Test1-SU2 (0-6)	248	230	173.6	yes	7.3
LSA Test1-SU3 (0-6)	1342	564	939.4	no	58.0
LSA Test1-SU3 (0-6)	986	387	690.2	no	60.8
LSA Test1-SU3 (6-24)	205	182	143.5	yes	11.2
LSA SUT 2,2 (0-6)	181	147	126.7	yes	18.8
LSA SUT 2-3,03 (0-6)	1726	1319	1208.2	yes	23.6
LSA SUT 3,O4 (0-6)	58.1	56.4	40.67	yes	2.9
LSA SUT 4,1 (0-6)	163	125	114.1	yes	23.3
LSA SUT 5,1 (0-6)	144	122	100.8	yes	15.3
LSA SUT 6,1 (0-6)	151	119	105.7	yes	21.2
LSA SUT 6-7,1 (0-6)	132	120	92.4	yes	9.1
LSA SUT 8,O2 (0-6)	155	97.3	108.5	no	37.2
LSA SUT 9,1 (0-6)	87.8	64.6	61.46	yes	26.4
LSA SUT 10,2 (0-6)	69.5	57.5	48.65	yes	17.3
LSA SUT 11,2 (0-6)	302	212	211.4	yes	29.8
LSA SUT 11,3 (0-6)	43575	1017	30502.5	no	97.7
LSA SUT 11,4 (0-6)	118	52	82.6	no	55.9
LSA SUT 12,I2 (0-6)	82.1	58	57.47	yes	29.4

Decision units (DUs) were planned as confirmation of narrow rectangular segments that would compose the 200-mg/kg lead boundaries around the LSA contaminated area, after SU data indicated the likely location of that border. Quadrangular DUs were used to characterize contamination around the EBA buildings. In order to manage decision uncertainty stemming from data variability, decisions about whether individual DUs exceed an action level were made based on the DU's 95% upper confidence limit (UCL). The calculation of this UCL was based on replicate field samples. Since the UCL calculation was made at a higher data level (i.e., field sample replicates), the mean of replicate bag XRF readings (not the bag UCL) was used as the "sample result." Two DUs were sampled in triplicate to assess field sample precision and representativeness.

Predicted UCLs were calculated for DUs having only a single ISM field sample (a "singlet DU"), using statistical inputs from other DUs. This strategy was used to maintain statistical decision confidence while reducing the unsustainable workload that would have resulted if all EBA DUs had triplicate ISM field samples collected. The strategy used to develop, check and implement UCL prediction for EBA DUs having only a single ISM sample ("singlet DUs") is described below. The first two EBA DUs collected [EBA DUs 1 (0-6) and EBA DU 2 (0-6)] both had triplicate ISM field samples, from which statistics were calculated. The averaged variability (as pooled RSD) from those two DUs was used in the UCL equation to calculate predicted UCLs for singlet DUs. The next two EBA DUs collected were called "check DUs" [EBA DU 10 (0-6) and EBA DU 10 (6-24)] and also had triplicate ISM samples so that actual UCLs could be calculated and "checked" against UCLs predicted for them based on the pooled variability derived from the first two DUs.

Table 3-4 below shows the comparisons between the predicted and actual UCLs for the two "check DUs." Note that a UCL is predicted from each sample result in the triplicate set for a check DU; each predicted UCL is compared with the actual UCL from the triplicate set using RSD as a measure of

difference. The differences between the predicted and the actual were all less than 10% RSD. At the 200 mg/kg threshold of interest, the range of ±10% RSD around a 200 mg/kg UCL = 174 to 230 mg/kg. This is an acceptable degree of decision uncertainty. The predicted 95% t-UCLs for DUs with single ISM samples are listed in Table 3-5 below. Single ISM results greater than 168 mg/kg would yield a predicted 95% t-UCL greater than 200 mg/kg.

Table 3-4: Comparison between the predicted and actual UCLs for the "check DUs"

Predicted UCL value calculation for single DU-IS samples for EBA check samples							
		Check DU	Pred 95% t-UCL			Diff between Pred & actual UCLs	
Enter the following value for single DUs:	DU ID	ISM Sample Result	[df = 5; n = 1]				as RSD
Check DU (using 1st Rep result)	DU10 (0-6)	328.0	389.1	actual t-UCL from DU10 (0-6) trip set =	385.8		0.6%
Check DU (using 2nd Rep result)	DU10 (0-6)	355.5	421.7				6.6%
Check DU (using 3rd Rep result)	DU10 (0-6)	368.8	437.5				9.5%
Check DU (using 1st Rep result)	DU10 (6-24)	129.3	153.3	actual t-UCL from DU10 (6-24) trip set =	154.7		0.6%
Check DU (using 2nd Rep result)	DU10 (6-24)	121.6	144.3				4.7%
Check DU (using 3rd Rep result)	DU10 (6-24)	147.1	174.5				9.0%

Table 3-5: Predicted 95% t-UCLs for DUs with single ISM samples

	Singlet EBA DU ISM Sample Result	Pred 95% t-UCL [df = 5; n = 1]
EBA DU2 (6-24)	46.5	55.2
EBA DU3 (0-6)	325.5	386.2
EBA DU3 (6-24)	206.8	245.4
EBA DU4 (0-6)	186.5	221.3
EBA DU4 (6-24)	67.6	80.2
EBA DU5 (0-6)	782.2	928.0
EBA DU6 (0-6)	257.7	305.7
EBA DU6 (6-24)	56.1	66.6
EBA DU7 (0-6)	180.8	214.4
EBA DU8 (0-6)	319.5	379.0
EBA DU9 (0-6)	1202.6	1426.6
EBA DU 11 (0-6)	151.9	180.1
EBA DU 12 (0-6)	102.4	121.5
EBA DU 13 (0-6)	161.6	191.7
EBA DU 14 (0-6)	178.1	211.3
EBA DU 15 (0-6)	150.9	179.0
EBA DU 16 (0-6)	385.8	457.7
EBA DU 17 (0-6)	400.9	475.6

3.5.2. Sample Processing QA/QC

Samples were processed by air-drying (if needed), and manual disaggregation (by hand crushing or with a small PVC roller). Sample sieving to 10-mesh (2 millimeters) was done when necessary to remove non-soil coarse material. The purpose of sample processing was 2-fold:

- Reduce within-sample variability, which generally reduces the number of replicate bag readings required to achieve sufficiently precise analysis. Sufficient precision is monitored by real-time calculation of within-sample %RSD and bagged sample confidence intervals.
- Unless otherwise specified on a site-specific basis, the <10-mesh soil fraction is considered representative of "soil" for the purpose of chemical analysis.

After processing, samples were placed in "read" bags (plastic bags verified with negligible interference with the XRF's X-rays) for placement on the stage of the XRF benchtop stand. The accuracy of reported sample bag concentrations was ensured by replicate analyses over sample bag contents. "Bag

replicates" include at least four sequential readings with the bag moved between readings to expose new portions of the sample for analysis. Real-time statistical evaluation of replicate bag readings provide a measure of subsampling precision (which is a measure of within-sample heterogeneity), and indication of how many bag readings are needed to achieve statistically confident results. All XRF readings were entered into the RTeX tool which provides real-time calculations, and serves as a record of results for future consultation and reports (Figure 3-11).

Figure 3-11: Example RTeX form for an EBA sample

A statistical summary of within-sample variability (as %RSD for replicate XRF readings on the same sample bag) for the bulk (non-sieved) and <10-mesh (sieved) samples are given in the Table 3-6 below. As expected, the within-sample variability for sieved samples had somewhat less "subsampling" variability as non-sieved samples. Nonetheless, the difference was not substantial, which speaks to the thoroughness of the disaggregation process for this site's soil matrices. Bulk samples sometimes required more XRF readings per bag than sieved samples. However, extra readings did not add significant workload since 15-second readings were used to analyze samples. Extra readings to control for within-sample variability was much less time- and labor-intensive than sieving samples.

Table 3-6: Statistical summary of within-sample variability

Summary for Within-Sample %RSD for Replicate XRF Bag Readings	Disaggregated, not-sieved bulk (as %)	Disaggregated, sieved, <10-mesh (as %)
RSD range for all bagged samples	0.06 - 176	2.3 - 73
Mean / median RSD for all bag samples	17.1 / 10.6	11.0 / 8.0

3.5.3. Laboratory Analysis QA/QC

3.5.3.1. Instrument Bias

Verification of the TIIB Niton XRF's lead calibration before the Wilcox project found the calibration biased low by 5% for standardized soil certified reference materials (CRMs) at concentrations up to 1400 mg/kg (Figure 3-12). Up to 300 mg/kg, the results of CRMs were biased low by 4%. In other words, a true concentration of 200 mg/kg (the project's action level) would be reported no lower than 192 mg/kg, on average. This level of bias around the 200-mg/kg project action level is considered acceptable because other decision uncertainty management mechanisms (such as the use of UCLs) more than compensate for uncertainty when sample concentrations far very near the action level (Figure 3-13).

The XRF was linear at least to 6000 mg/kg lead (the highest value of available CRMs). A few samples had concentrations into the tens of thousands, (one as high as 98,000 mg/kg by XRF). No doubt such results are biased low by some unknown amount, since the XRF response is expected to fall off and become nonlinear at such high concentrations. However, the information value provided (i.e. the sample exceeds the screening level) is no different than if an unbiased result could be reported (Figure 3-14).

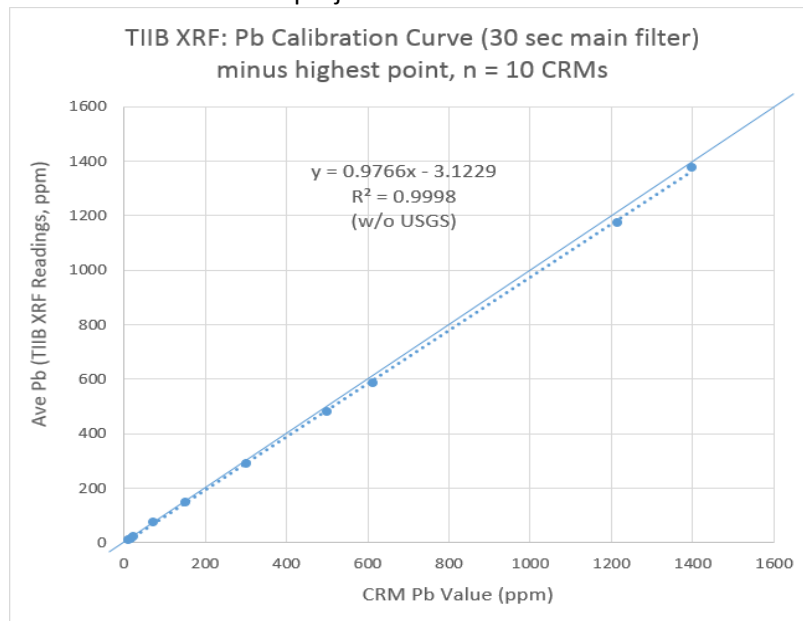Figure 3-12: Calibration before the Wilcox project

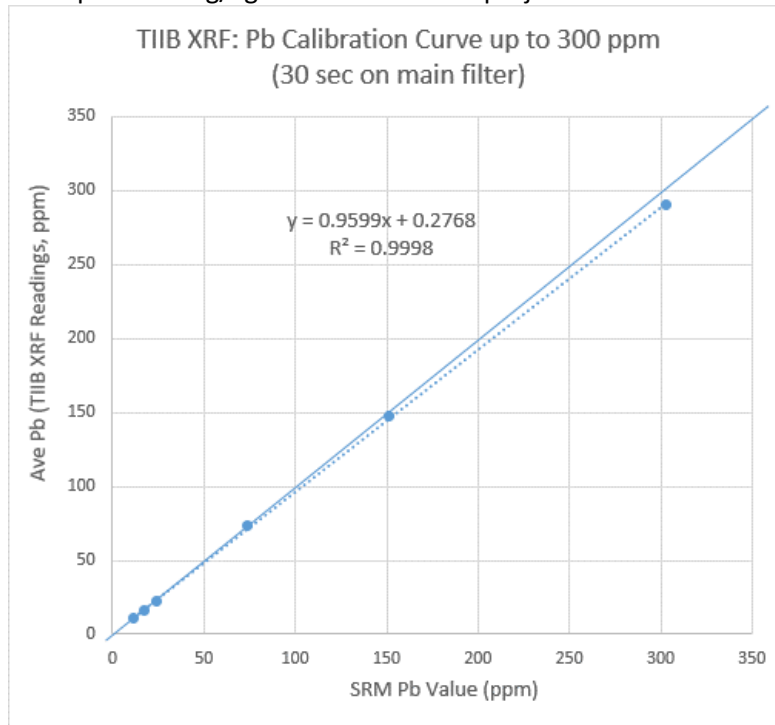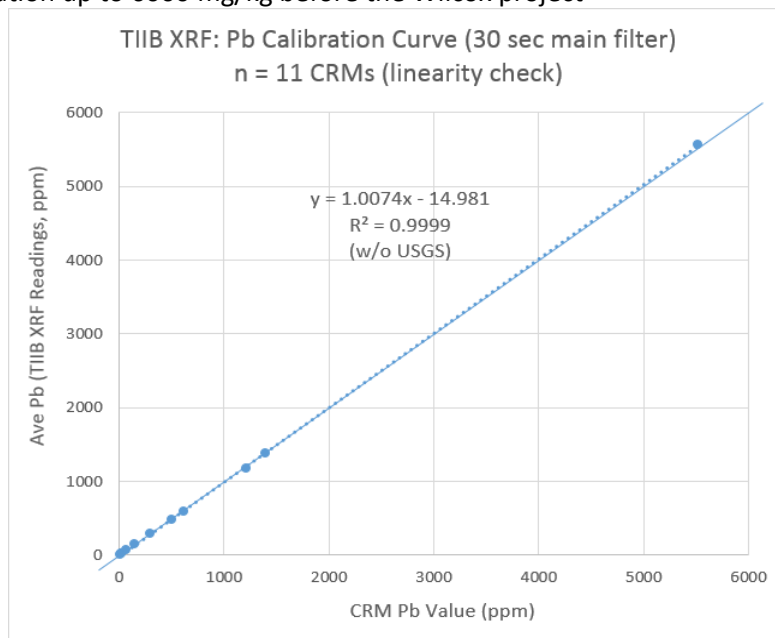Figure 3-13: Calibration up to 300 mg/kg before the Wilcox project

Figure 3-14: Calibration up to 6000 mg/kg before the Wilcox project

3.5.3.2. Instrument Precision

CRM precision at two different XRF read times is given in Table 3-7 below (CRM cups moved between readings). As expected, reading precision for CRM cups is slightly higher at the longer read time. However, this small increase in data imprecision is considered negligible for soil sample results, which were run at a read time of 15 secs.

Table 3-7: CRM precision at two different XRF read times

CRM lead Concentration	%RSD at 15-sec read time	%RSD at 30-sec read time
73	8.1	4.8
470	2.6	2.0
1370	1.7	1.4

To monitor XRF performance and validate sample data during deployment, three CRMs were selected as Laboratory Control Samples (LCS) and analyzed with the XRF. The LCS results are plotted on a control chart or a graphical representation of the acceptable limits for concentration results from a CRM of known concentration. The purpose of a control chart is to monitor the performance of an XRF instrument before and after batches of field samples are analyzed.

Three levels of CRMs were used as LCSs for the Wilcox project, JSAC 0462 (certified concentration = 73 mg/kg); RCRA (nominal concentration = 500 mg/kg); and NIST 2711a (certified concentration = 1370 mg/kg). Control charts were constructed using the most recent 30-second measurement data available. Because there was a lack of access to the XRF prior to the project, the initial data set included only 12-15 readings, about half of the number desired for control chart construction. The completed control charts for the October 25 to November 4, 2017 time period are shown in Appendix 3.

Low and high LCSs performed as expected, however the mid-range LCS (RCRA) had excessive variability, frequently exceeding ±2 SD limits, and occasionally exceeding ±3 SD limits. At the end of November 4, 2017, the original 12-15 LCS data was combined with the newly collected LCS data to recalculate new control chart limits. Original versus recalculated means and SDs are shown in Table 3-8. All LCS means were stable. The SDs were stable for the low and high LCSs but increased for the RCRA LCS. The performance with the new control charts for the low and high LCSs was still satisfactory, but performance of the RCRA LCS continued to be erratic (Appendix 3). Data will be collected on the RCRA LCS sample cup to monitor it, but it will not be selected for use as an LCS in future projects.

Table 3-8: Original versus recalculated means and SDs

Reference Materia ID	CRM lead Conc. (mg/kg)	Original values (n = 12 – 15)	Recalculated values (n = 36 – 42)
JSAC 0462	73	Mean = 73.6; SD = 3.34	Mean = 72.9; SD = 3.37
RCRA	470	Mean = 477; SD = 5.1	Mean = 476; SD = 8.1
NIST 2711a	1370	Mean = 1371; SD = 17.2	Mean = 1370; SD = 16.2

4. Conclusions and Recommendations

The primary project objective was to identify the spatial extent of lead with a concentration of greater than 200 mg/kg in the 0 to 6 and 0 to 24-inch intervals at the EBA and LSA to support risk assessment and mitigation decisions. Related objectives were to identify and delineate hot spots and if possible, estimate contaminated soil volume to support removal evaluations.

Secondary objectives stated in the QAPP included:

1. Introduce regional EPA staff to sampling, processing and analysis techniques for Incremental Composite Sampling and XRF to rapidly and definitively identify the concentration of lead in soil. Field methods, sample preparation procedures and analytical procedures, will be evaluated as part of the study.
2. Evaluate the relative distribution of lead among soil particle size fractions for a few select samples. Determine whether higher lead concentrations in finer particles are more likely to contribute to storm run-off to streams. Evaluate the particle size and lead content of any crystalline material observed in the LSA to determine whether soil sieving at 100-mesh could remove this material and bias soil results. Retain samples of any crystalline material for potential laboratory evaluation of water solubility and/or other relevant characteristics.
3. Comparability evaluation for the two XRFs to be used on the project so that the lead results from the XRF units can be used interchangeably.
4. Evaluation of the field, sample prep, XRF analysis, and statistical analysis methods used for this project to improve the ICS/XRF methodology. The results of this analysis are not included here, and will be provided to EPA OSRTI TIIB under separate cover.

4.1. Lead Distribution at the EBA

Project objectives were only partially met at the EBA because definitive boundaries of lead with a concentration greater than 200 mg/kg in the 0 to 6 and 0 to 24-inch intervals were not identified. However, several areas were definitively identified as exceeding the screening level. These areas covered 10,227 square feet in the shallow and 3,660 square feet of soil in the deep DUs and represented 189 cubic yards of soil in the shallow and 68 cubic yards of soil in the deep DUs. The sampling did identify the approximate contaminated area for one PSA.

Shallow (0 to 6-inches) lead contamination above the 200 mg/kg screening level was found throughout the sampled area at the EBA (Figure 3-3) and likely extends beyond the area sampled to the south and east (Inset A). While the thin strip of soil between the northwest building and the fence (Inset B) were, and DU 2 at the northeast corner of the building (Inset C) were below the screening level, all other areas adjacent to the building exceeded the screening level. The dirt floor within the southeast building exceeded screening level, but there was a small area beneath the concrete floor near the western wall

could not be sampled. The west building was unsafe for entry and appeared to have a concrete slab floor so no samples were collected with the building.

DUs 2 and 11 in the northeast side of the EBA buildings (Inset C) did not exceed screening levels, but the elevated concentration trends in DUs 14 and 17 to the south (Inset A), and the presence of an access road and historic operations east of the DU do not support a conclusive demonstration that the boundary of lead contamination at the EBA is defined by the results of DUs 2 and 11.

SU samples from the access road to the north adjacent to Distillation Process Area 1 had concentrations above screening levels, but DUs 12 and 15 to the west and south were below the screening level (Inset D). The heavily wooded area between the road and fence contained two concrete structures (possibly foundations or shallow basins) and a large amount of metal debris. Historic and aerial photos indicated that this area was open and contained two small structures but did not appear to contain any processing equipment or storage. It would be reasonable to assume that DUs 12 and 15 represent a locally uncontaminated area outside the largely contaminated EBA area. DU 13 was created to evaluate the north side of the concrete pad north of the EBA and adjacent to Distillation Process Area 1. The results indicated that the 0 to 6-inch soil in the DU is below the screening level (176 mg/kg), but still relatively high.

The near surface material (0 to 3 inches) from the two ramps leading to the southeast building was sampled and both exceeded the screening levels. The ramps appeared to be built up to building floor level from the ground surface using a gravel size material, but the nature of the construction material was not investigated.

The lead contamination in the PSA and DUs adjacent to the buildings is likely related to the historic activities at the EBA, however it is unclear if the contamination at more distant DUs is related to the EBA or to roads, storage areas, and adjacent historic refinery operations. The preliminary CSM assumed that contamination at the EBA was sourced from the operations in and near the buildings and decreased to low levels away from the source. Results for the sampling did not fully support this CSM. Step-out DUs to the south and southeast of the EBA did not identify a 200 mg/kg boundary and showed a decreasing concentration but then increased further away from the building. The more distant DUs appear to overlap with the access road from the former Steam Plant, material storage areas near the rail siding, and Distillation Process Area 2. The asphalt-like material found at DUs 5, 10, 7, and 14 and in the unsampled areas south of these DUs suggests that contamination at these areas may not be directly related to the EBA process and reflect the road materials and process waste high in lead. A 200 mg/kg boundary could not be identified to the south

and southeast and it is likely, based on the overlapping LSA samples in the area, that these areas could exceed 200 mg/kg.

Contamination in the 0 to 24-inch interval was identified on the north and south sides of the buildings at DUs 3 and 10, and at SU 2. DU 3 appeared to contain a large amount of debris within a concrete structure and exhibited several increments with oily soil at depths greater than 12 inches (Figure 4-1). Similarly, the 0 to 24 intervals from SUT 1,1 and SUT 1,3 north of the site also exceeded the screening level and had oil odors and oil-stained soil. A 6 to 24-inch DU was not developed under the shallow DU 5 south of the building because the deep sample from SU 2 suggested the deep DU there would exceed the screening level. The deep step out at DU 10 exceeded the screening level, however no additional DUs were developed south of DU 10 due to time constraints.

Deep soils (0 to 24-inches) at the EBA are above screening levels in areas to the north and south of the buildings (Figure 3-3), but a clear 200 mg/kg boundary line could not be established. Deep contamination likely extends to the north of DU 3 under the road, and to the south of DU 10 along the rail siding. Given the proximity of other historic activities (former Steam Plant and Distillation Process Areas 1 and 2) DUs 1 and 6 may represent local areas below the 200 mg/kg screening level that do not continue east.

The sampling approach for the EBA, including PSA identification, SU and DU samples, and core segment analysis was effective in developing an understanding of the distribution of contamination in the shallow and deep soil segments. An important aspect of the approach was creating appropriately sized DUs with similar characteristics. DU sizes ranged from 129 to 4693 square feet, and effectively represented the conditions of each area. The rigorous quality control provided a high level of certainty to the sample results, however the effects of adjacent source areas and roads do not allow a definitive statement that areas outside of the contaminated DUs are less than 200 mg/kg.

Figure 4-1: Photo of 0-24-inch soil core with a black oily substance found in EBA DU-3.

4.2.   Lead Distribution at the LSA

Project objectives were only partially met at the LSA because a definitive boundary of lead with a concentration of greater than 200 mg/kg in the 0 to 6 and 0 to 24-inch intervals was not identified. The study identified large areas with concentrations greater than the screening level of 200 mg/kg. Within the 10-acre area studied, up to 76 percent (6,275 cubic yards) was interpreted by geostatistical analysis as potentially exceeding the screening level in the shallow soil. In the deep interval sample results defined a minimum volume of 6,149 cubic yards above the screening level.

Shallow Lead Distribution.

Shallow (0 to 6-inches) lead contamination above the 200 mg/kg screening level was extensive throughout the LSA and extends to the northwest and northeast beyond the LSA area. Figure 3-5 shows the results of SU samples and DUs, and Figure 4-2 is a kriged isopleth map showing the interpreted areas with concentrations of lead at 200, 400, and 1,000 mg/kg. Samples from areas east, south and southwest of the main source area showed concentrations less than the 200 mg/kg screening level, however confirmatory samples demonstrate a low level of confidence that these samples represent a true boundary of the contaminated area. The soil in the LSA area was extensively reworked in several areas and there was a large amount of metal and broken concrete debris as well as several intact concrete structures.

SU samples from radial transects 3 through 6 on the east side Inset E demonstrated a decreasing trend of contamination outward from the source area as hypothesized in the preliminary CSM. An area less than 200 mg/kg was roughly bounded by the existing pond and inlet stream (Figure 4-2). Samples immediately east of the pond had lead results below 200 mg/kg, however the DUs developed to confirm the 200 mg/kg boundary (DU 5-6 and DU 6-7 and DU 7-8) resulted in statistically confident values above the screening level. Samples LSA SUT 5,3 and LSA SUT 6,2, located approximately 100 feet east of the DUs, had concentrations below 100 mg/kg suggesting that a boundary may be present further east. Confirmatory DU 4-5 on the north end of the pond was the only confirmatory DU with a statistically confident concentration below the screening level. We conclude that the

area on the east side of the LSA between transects 4 and 7 where concentrations appear to be less than screening level could represent a local area less than the screening level, but a definitive boundary is not established because other source areas (ponds) appear to overlap and abut the eastern extent of contamination at the LSA.
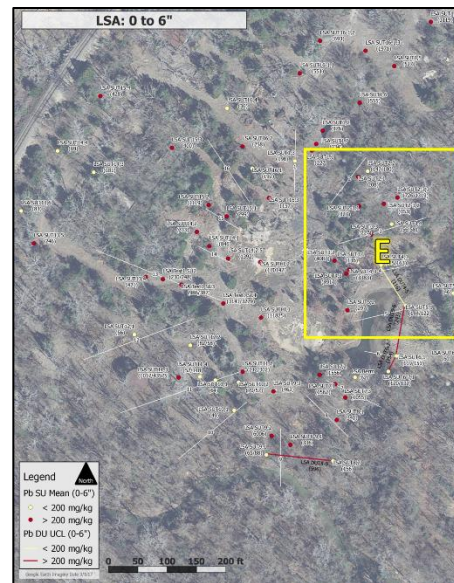
On the south side of the LSA, Transects 7, 8 and 9 (Inset F) are bounded by the deeply incised stream valleys for West Tributary and Sand Creek. At the top of the ravine, a high fence restricts access to the stream bank. A berm is present at the south end of the existing pond and a berm for former Tank 37 forms the southern side of the area between Transects 8 and 9. Samples were collected up to the fence at Transects 8 and 9, and one sample on Transect 7 (LSA SUT 7,3) was collected beyond the fence on the slope of the incised valley. The SU samples at the outer most points of Transects 8 and 9, located at the

fence, were the only samples less than the screening level and a DU along the berm was developed and sampled in triplicate. Results of the DU were considerably higher than the screening level (304, 332, and 547 mg/kg). Closer inspection of the bermed area identified a significant amount of reworked material on the edge of the berm that likely originated from the Tank 37 area. We attribute the significant difference in concentrations between the SU and DU samples at Transects 8 and 9 to the area represented by each sample type. SU samples represent very small areas (4 square feet) at the outermost edge of the berm, whereas the DU sample represented a 1,300 square foot area on top of the berm which likely received contaminated material from the LSA and Tank 37 area as a result of reworking and disturbance. While a statistically confident boundary was not established, the extent of contamination at the south end of the LSA is limited

by the physical edge of valley. However, results of sample SUT 7,3 (1,055 mg/kg) from the edge of the stream valley, suggest that contaminated material may have moved downslope.
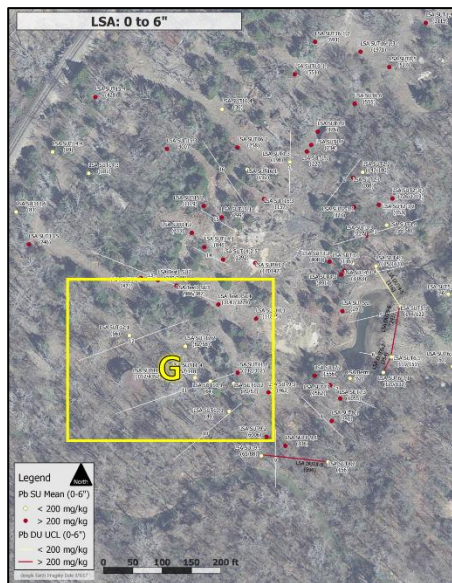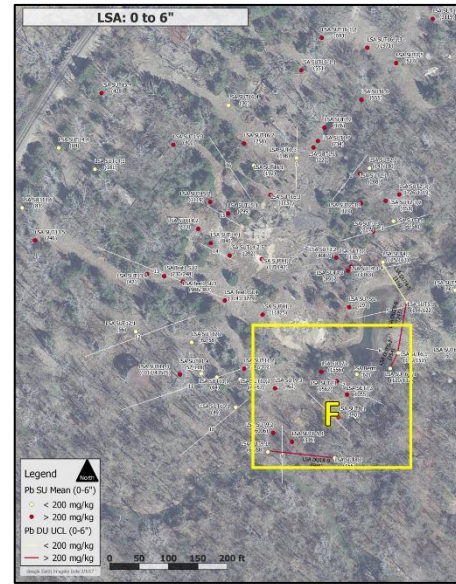
The outer SU samples on transects 10 through 12 on the southwest side of the LSA (Inset G) generally had results less than the screening level with the exception of SUT 11,3. The main access road and cut road north of Tanks 35, 36, and 37 mark the approximate boundary of the area that appears to be less than 200 mg/kg (Figure 4-2). SUT 11,3 was collected within the bermed area for Tank 35. It was initially thought that SUT 11, 3 would be collected to confirm the extent of contamination on Transect 11, however, the sample had a concentration of 1017 mg/kg and a duplicate sample had a concentration of 43,575 mg/kg. The high concentrations and variability suggested that the bermed tank area represented a different source that exceeded the screening level. To evaluate the heterogeneity of the soils in the bermed area, two grab samples were collected from light-colored soils in un-vegetated locations near SUT 11, 3. Both samples showed concentrations above the screening level (200 and 204 mg/kg), but lower than the samples form SUT 11,3. The kriged data map (Figure 4-2) shows a large part of the area between transects 10 through 12 as less than the screening level, however this is an artifact of the biased sampling approach used to reflect the preliminary CSM. We conclude that the area on the southwest side of the LSA where concentrations appear to be less than screening level as shown on the kriged data maps, does not represent a widespread area of concentrations less then screening level and likely represents a different historic use (Tanks 34, 35, 36, as shown by the red outline from ERT Sheet 1A) that overlaps or abuts contamination derived from LSA source. A definitive boundary of contamination less than the screening level was not identified in the southwest part of the LSA.

Transects 13 through 2 (Inset H) cover the northern part of the LSA where concentrations exceeded the screening level at most sampled locations. Samples with concentrations less than the screening level were broadly distributed and did not define a clear boundary. At transects 13 and 14, the three samples with concentrations below the screening level were located 200 to 350 feet from the main access road (assumed to be the physical boundary of lead sweetening operational area), and within other former process areas. Two of the samples were located near the Wilcox Refinery western property boundary fence and one sample was located within the former Distillation Process Area 2. Soils in these areas appeared to be reworked as evidenced by debris, uneven ground, and localized mounds of soil and roots. Sample SUT 16,4, which had a concentration of 36 mg/kg, was located adjacent to a cut road in and may represent reworked deeper soils. Samples SUT 16,1 and SUT 1,3 appear to be localized spots with concentrations just below the screening level (192 and 198 mg/kg). Transects in the northern part of the LSA were designed to traverse from the highest concentrations in the process operations area outward to areas less affected by the LSA operations. Most transects demonstrated decreasing concentrations away from the source area, however, the trend did not continue to decrease below screening level, and in many cases it reversed and elevated concentrations were encountered moving further from the LSA source and into overlapping or abutting source areas. For example, outer samples at Transects 16 and 1 are clearly within the Upper Process Area (Distillation Process Area 1) and the Product Storage Area (Tanks 22 through 30), and Transects 2 and 3 cross the berm for the former cooling water/makeup water pond. Predominant wind direction is from the south and southeast, with secondary wind direction from the north (USDA), which may partially explain the broader distribution of lead to the north and south because finer grained materials tend to have higher lead concentrations and are more likely to be transported by wind. A 200 mg/kg boundary is not established in the northern part of the LSA because other lead source areas appear to overlap and abut the contamination at the LSA.
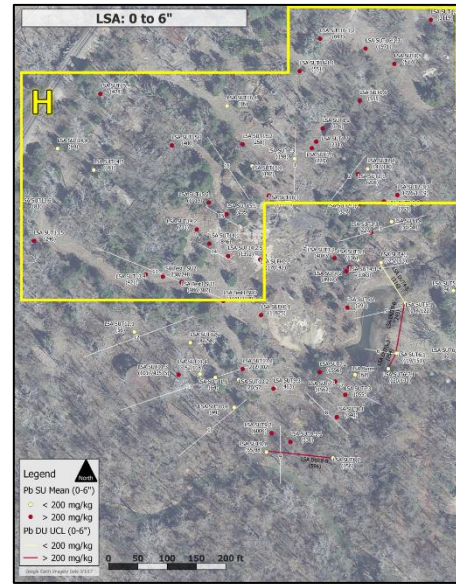
Overlapping and adjacent sources are present in the study area as shown in old air photos. Figure 4-3 is an overlay of the kriged lead concentrations on the 1956 aerial photo. The photo was annotated for the ERT report (Lockheed, 2016) to show various process and storage areas. The photo clearly identifies LSA samples that are located in former operational and storage areas.

In summary, at the LSA a definitive boundary identifying the area of 0 to 6-inch soils above the screening level cannot be confidently identified because:

1. Other lead sources overlap or about the LSA impacted area at concentrations greater than the screening level,
2. There has been extensive re-working of near-surface soils during post refinery operations, and demolition, causing mixing and redistribution of lead-contaminated soils, and
3. Roads constructed of or exposing material that is not representative of the LSA soils cut through the LSA and act as transport mechanisms and secondary source areas.

Deep lead distribution.

Deep soil samples (0 to 24-inches) were collected at twenty locations to help define the extent of lead in the subsurface (Figure 4-4). Only one sample (LSA SUT 2,1) had a concentration less than the screening level. The highest concentrations were found in the south-central part of the LSA in un-vegetated areas.

Ten of the deep samples were segregated into 4-inch depth intervals to evaluate the vertical distribution of lead. Figure 3-7 summarizes the results of the profiles. The two samples in the center of the LSA where the ERT study (Lockheed, 2016) identified percent-level concentrations on the surface, and the two samples from the eastern side of the LSA all show concentrations greater than 1000 mg/kg throughout the 0 to 24-inch section. Sample LSA SUT 2,3 also showed lead contamination above the screening level throughout the 24-inch interval. Soil samples on Transect 9 indicated the potential for a hot spot near LSA SUT 9,2 so three deep samples were collected to the north, south, and east to evaluate the extent of the high readings. The samples indicated deep contamination was present throughout the area and may represent a localized deep hot spot. Although the remaining samples all exceeded the screening level for the entire core, the deeper intervals generally showed concentrations below the screening level.

The 0 to 24-inch data suggests that the deep contamination is likely bounded on the northwest by shallow bedrock which was encountered just east of the main access road at Transect 15, and extends to the south to the berm on the end of Transect 9. The limited number of samples and distribution of samples with concentrations less than the screening level precluded geostatistical analysis to define a 200 mg/kg boundary, however the sample results define a minimum area of 1.9 acres above 200 mg/kg. ERT boring data indicates that the lead contamination extends below 4 feet at LSA SUFHi,2 location (ERT sample WIL AA-10), but is contained within the upper 1 to 2 feet at three other deep sample locations.

Figure 4-2: Kriged isopleth map showing the interpreted areas with concentrations of lead at 200, 400, and 1,000 mg/kg (2017 aerial photo)
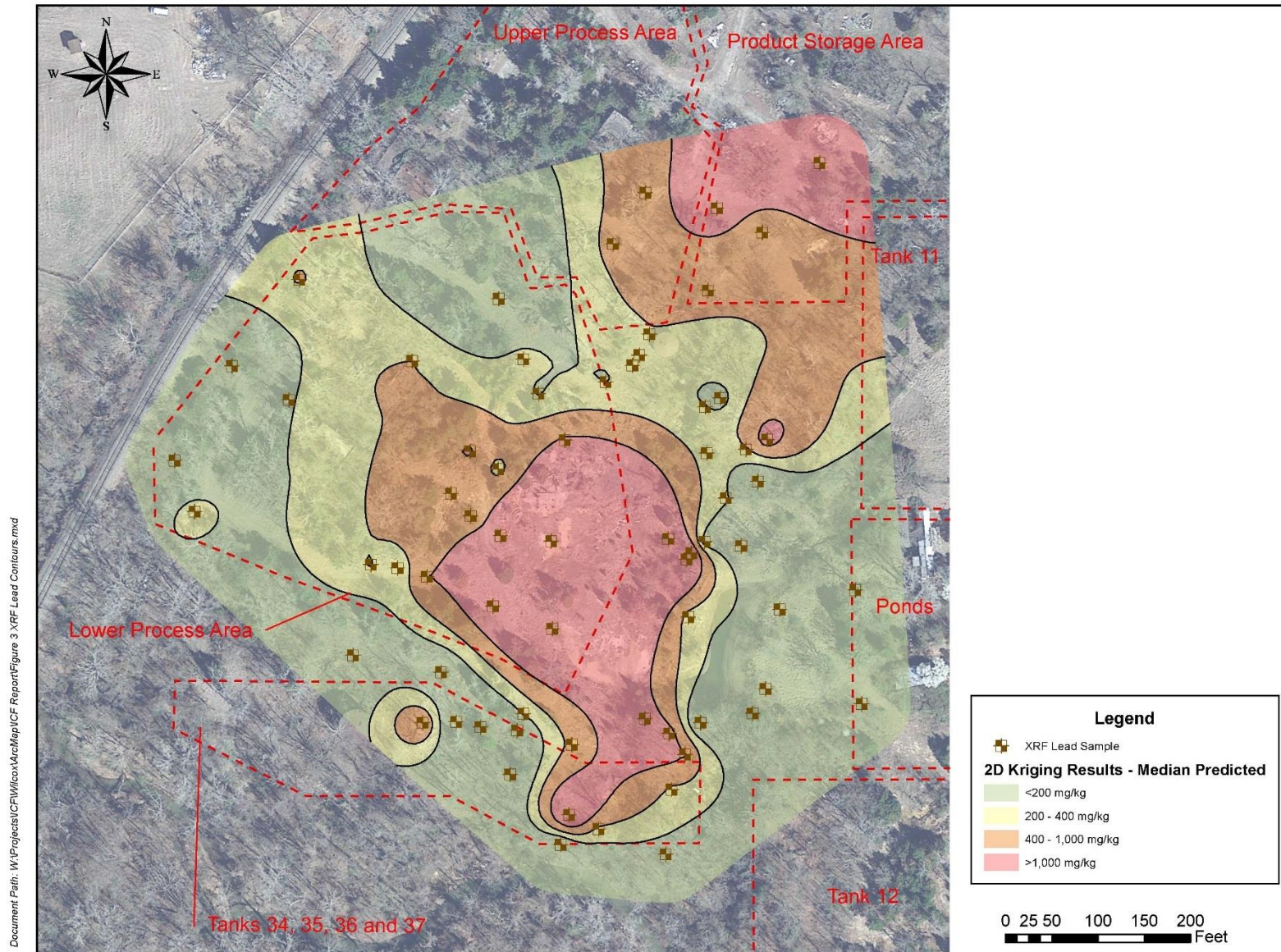
Figure 4-3: Kriged isopleth map showing the interpreted areas with concentrations of lead at 200, 400, and 1,000 mg/kg (1956 aerial photo)
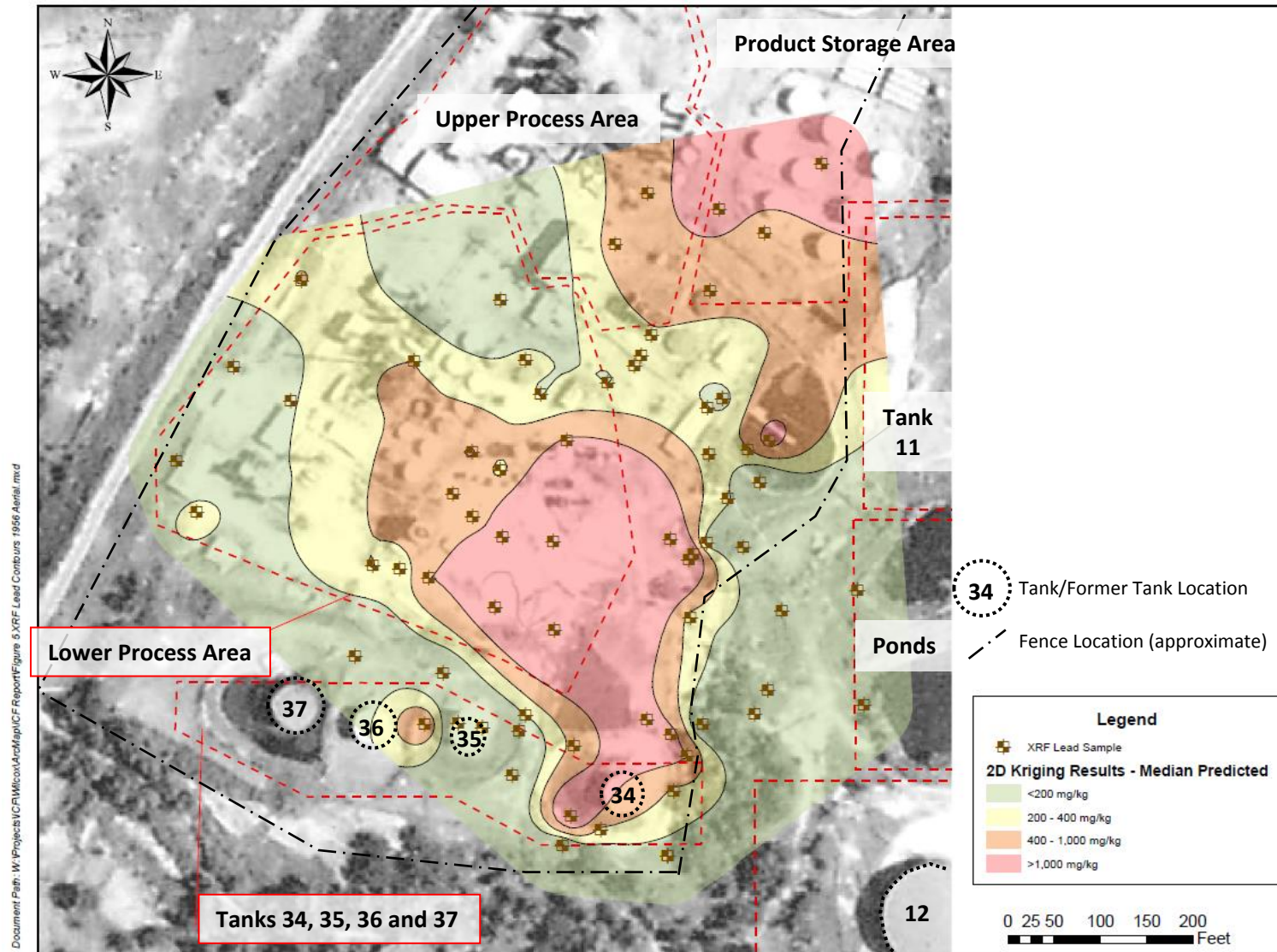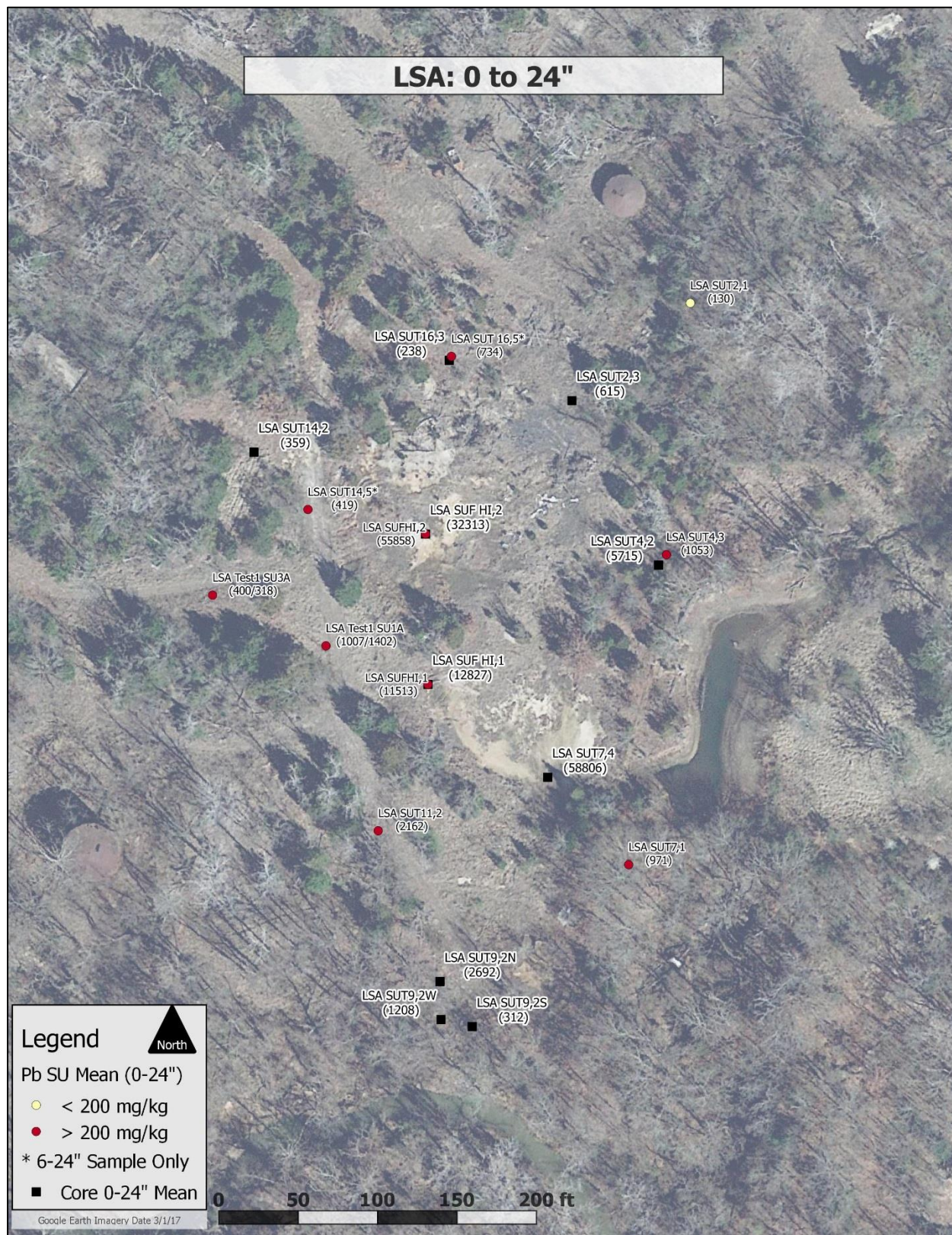
Figure 4-4: LSA 0 to 24-inch sample locations and results

4.3. Lead Distribution on Roads

Results of the nine road and berm samples suggest that the main access road and the other built roads contain high lead concentrations that may act as sources if the road material is transported to other site areas (Figure 3-8). It is unknown if the original construction material contained lead or if the roads became contaminated as a result of transport from the LSA. Cut roads have lower concentrations than the built roads, perhaps because they are further from the LSA source area and represent more recent transport of lead-contaminated materials during post refinery activities. Berm surfaces appear to be constructed of material with low lead content or were not used as vehicle pathways from contaminated areas.

4.4. Comparison with ERT XRF screening

At the EBA, the 200 mg/kg contour line generated from the ERT kriged data (Lockheed Martin, 2016) did not match well with the results of the DU and SU results. Handheld XRF results from south and east of the building suggested much lower concentrations than the 0 to 6-inch DUs in the same areas. The result was a significant underestimate of the area of contamination by the handheld XRF. This is likely attributed to the very shallow depth of investigation of the XRF and the heterogeneity of the surface materials. The areas less than 200 mg/kg near the fence north of the buildings were not detected due to limited spatial distribution of sample points in the ERT screening. The result was a significant overestimate of the area of contamination by the handheld XRF in the area north of the EBA building.

Identification of the highest concentration areas in the center of the LSA was generally consistent between the ERT kriged data and the ICS investigation. The quantification of lead concentrations, however, was not consistent because of the difference in the two methods. The handheld XRF had a limited area of measurement and depth penetration (essentially a point at the ground surface) and the SU samples included a homogenized volume of soil from the top 6-inches over a 4 square foot area. Lead concentrations in samples from the center of the LSA analyzed by the handheld XRF were much higher than corresponding SU samples and reportedly contained high lead content crystal grains. Also, at high concentrations, all samples were well above the effective calibration range of the XRF instruments. The 80% confidence maximum predicted 200 mg/kg from the ERT results was generally consistent the 200 mg/kg areas on the east side (Transects 4, 5 and 6) and the southeast side (Transects 10 through 12) but not consistent in other areas of the LSA (Figure 4-5). We attribute much of this inconsistency to the limited extent of the sampling points used to develop the ERT 200 mg/kg boundary and the limitations of geostatistical analysis for kriging the biased data set. The larger spatial distribution of samples in this study was driven by the CSM and resulted in geostatistical analysis incorporating adjacent and overlapping lead contamination from areas outside the LSA operational area.

Figure 4-5: LSA 0 to 6-inch 2-D Kriging results with ERT 200 mg/kg median predicted extent
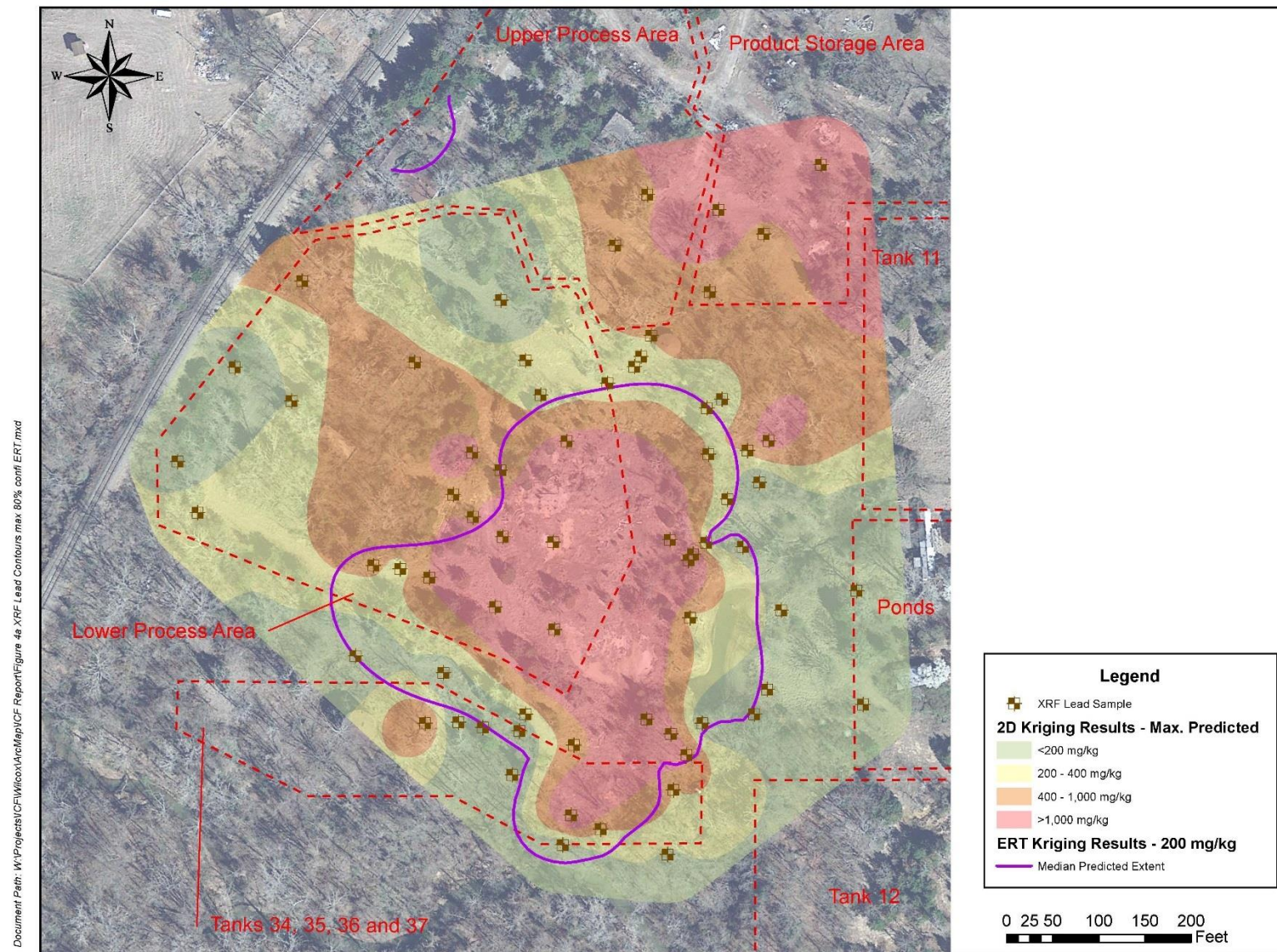
4.5.   Revised CSM

Data collected during the adaptive sampling program led to real-time revision of the preliminary CSM as described in the sections above. The revised CSM recognizes that high levels of lead contamination exist throughout the Wilcox area from many former operations across large areas. Lead particles transported by re-worked shallow material, wind, and vehicle travel further expanded the affected areas such that areas with concentrations less than 200 mg/kg are relatively small and represent the exception rather than the base condition. The main access and cut roads are contaminated and act as sources for further dispersal of contamination. Soils in several areas are highly disturbed which has mixed lead particles with natural and placed materials. The scale of heterogeneity of soils necessitates a decision-unit based incremental sampling approach for decision making.

Figure 4-6, Figure 4-7, and Figure 4-8 illustrate the conceptual model for overlapping sources and the impact of sample density and screening level on interpretation. In Figure 4-6 the preliminary CSM from the QAPP is represented by the separation of the LSA and nearby Source A. The preliminary CSM assumed that the LSA was spatially separated from other sources and the concentration gradient (i.e. source influence) would decrease outward from the LSA source to a value less than the screening level, where a definitive boundary could be identified. In the preliminary CSM, the influence of lead concentrations above the screening level from Source A terminates when concentrations reach the screening level and Source A is spatially separated from the LSA source. The revised CSM, based on sample results, observations in the field, and historical photographs and maps, is illustrated in the relationship between the LSA and Source B (Figure 4-6). The concentration gradient from the LSA source overlaps the concentration gradient from Source B and a distinct screening level boundary cannot be identified.

Figure 4-7 demonstrates the effect of selecting an alternate higher screening level that would allow for the LSA and Source B areas boundaries to be spatially separated. Raising the screening level could mask the effects of smaller sources as shown by Source A. Sample density plays an important role in interpreting the extent of a source area as demonstrated in Figure 4-8. Widely spaced sample points could either miss sources (Source A) or be misinterpreted as a continuous gradient connecting two unrelated source areas (Source B). In this study, closely spaced sample points, selected using real time data and an adaptive sampling design, were able to identify the influence of adjacent source areas and improve the CSM.

The effect of overlapping sources is illustrated in the cross sections shown in Figure 3-8. Cross section A-A' does not appear to encounter any major source areas other than the LSA and demonstrates high concentrations in the center of the LSA and concentrations below the screening level near the ends of the cross sections. Because the concentrations do not approach background levels (estimated to be in the 10 to 30 mg/kg range) some impact of other sources or transport mechanisms is suspected. The northern end of cross section B-B' indicates an increasing concentration gradient between LSA SUT 16,1 and LSA SUT 16,2 as these points move closer to the former product storage area. Points on cross section C may reflect a combination of LSA source material and tanks 34, 35, 36, 37 and 12.

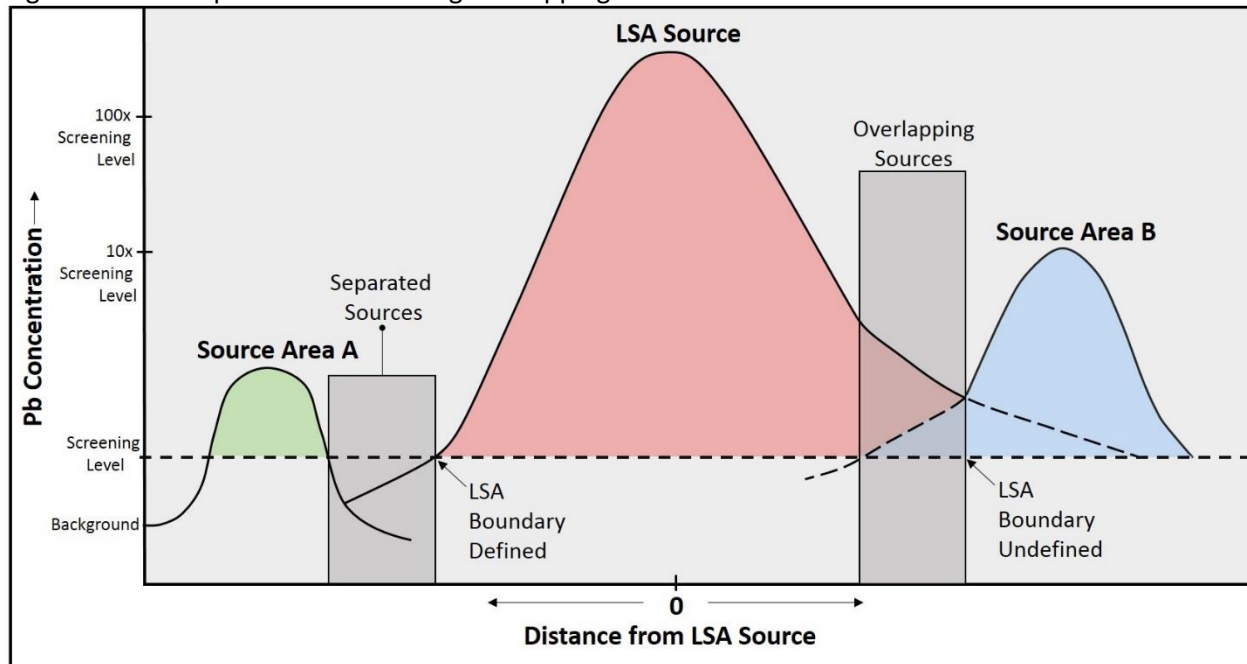Figure 4-6: Conceptual model showing overlapping sources

Figure 4-7: Conceptual model showing overlapping sources and screening level effects
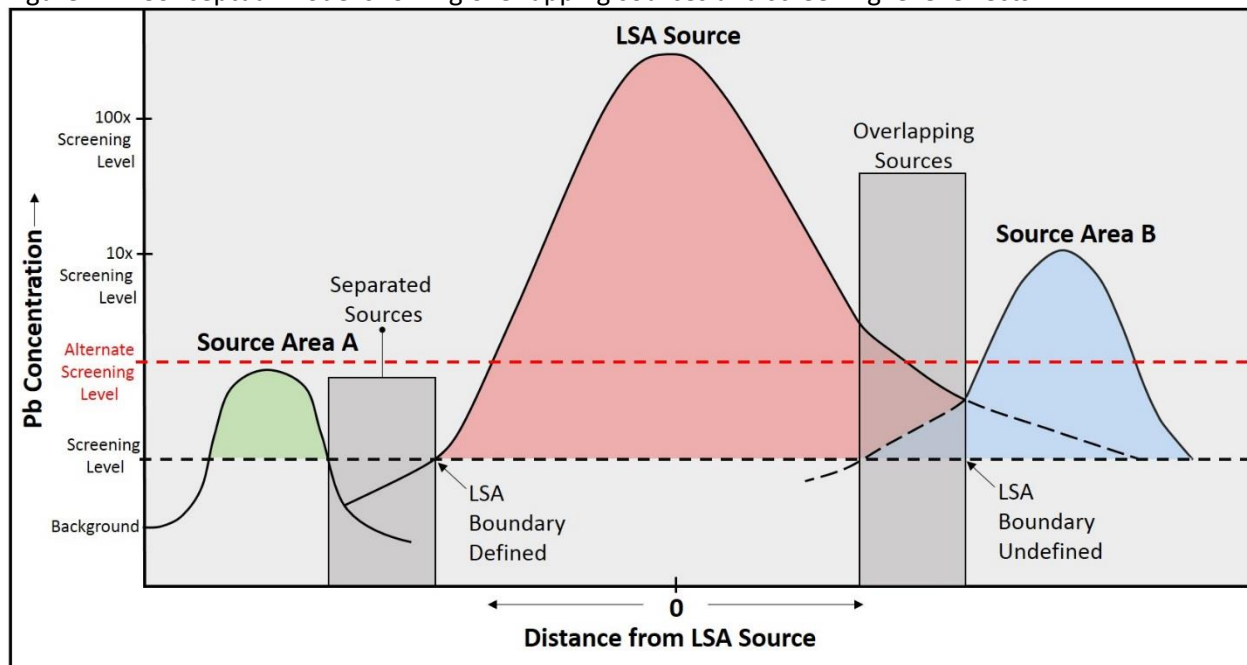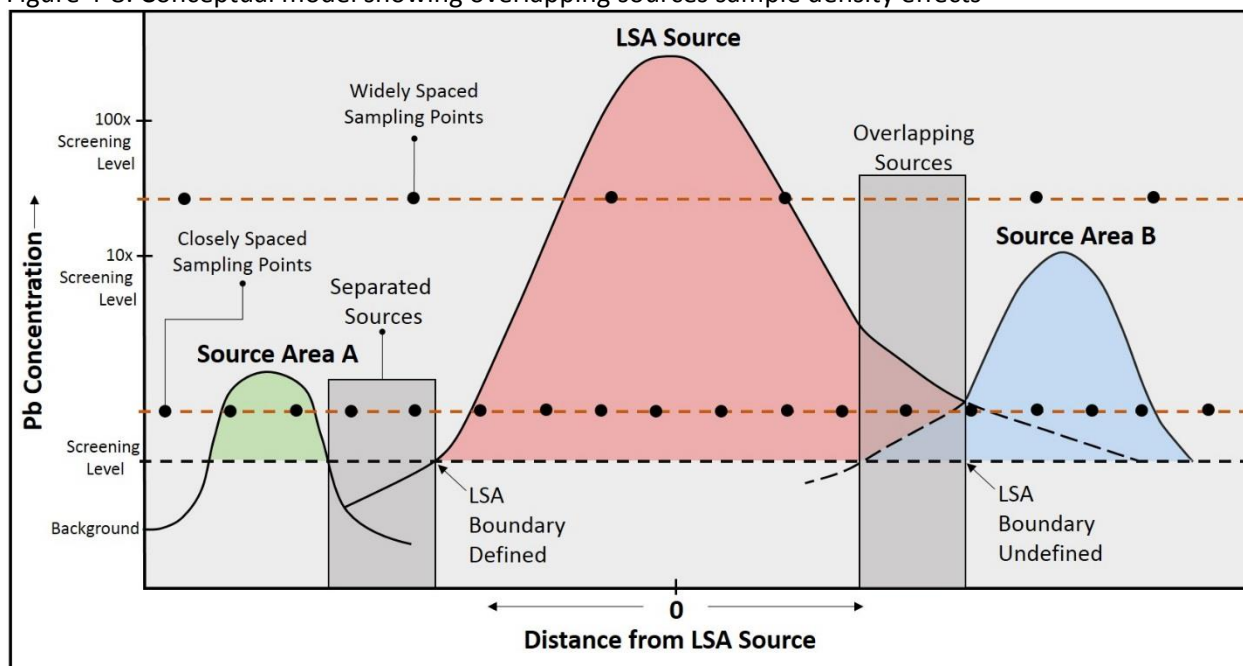
Figure 4-8: Conceptual model showing overlapping sources sample density effects

4.6. Secondary Objectives

Regional EPA staff and state counterparts were introduced to the sampling, processing and analysis techniques for ICS and XRF through frequent observation of field and laboratory activities, and discussion of the project status during execution. Daily field reports provided to the EPA Region and State project teams documented progress, results and challenges during the investigation (Appendix 5).

The lead concentration of the <125-micron particle size fraction in subsurface soils of the EBA appears enriched relative to the larger fractions. The soils at depth in this area had large amounts of very fine-grained material (silt and clay), that particles of which would reside in the <125-micron fraction and which would tend to bind more lead than larger particle fractions. However, the investigation found that lead concentrations do decrease with increasing depth. Surface soils in both the EBA and LSA show the same pattern of lead enrichment on the smallest particle sizes. Surface soils are more accessible to erosion by wind and water. Even mild breezes and minor rain events can mobilize very small particles exposed on bare soil, and these small particles carry a disproportionately larger amount of lead.

4.7. Recommendations

The sampling program generated a robust data set that is useful for project decision making, however decisions should reflect the revised CSM including overlapping lead source areas, main access and cut road contamination, highly disturbed soils, and lead particle transport via roads and wind. Specifically:

- An incremental sampling approach using CSM-based DUs should be used for any further soil characterization within the Wilcox Refinery Area. Existing results from discrete soil samples should be used with great caution in decision making. The highly heterogeneous nature of the lead contamination, makes it even more important to make remediation decisions that are backed by statistically defensible data.

- Travel on the main access road and certain cut roads should be eliminated until the roads no longer act as sources for dispersal of lead contamination. Vehicle travel on the main access road should be evaluated to determine if dust is transported offsite.
- The upper portion of the streambanks between Transects 7 and 9 should be further evaluated to determine if lead contaminated materials are being transported downslope.

A definitive boundary at the 200 mg/kg screening level cannot be definitively identified at the LSA and EBA because of the widespread contamination from overlapping and abutting source areas. A higher screening level may allow more definitive delineation of contaminated areas. The sampling strategy used in this study was based on the 200 mg/kg screening level, so samples may not be optimally placed to define contaminated areas at higher screening levels.

The finer grain size fraction should be analyzed for use in exposure and risk assessment analysis.

5.　　References

EA Engineering, Science, and Technology, Inc., PBC (EA). 2016. Draft Sampling and Analysis Plan for Remedial Investigation and Feasibility Study for Wilcox Oil Company Superfund Site, Bristow, Creek County, Oklahoma. April.

EPA. 2016. Recommendations for Sieving Soil and Dust Samples at Lead Sites for Assessment of Incidental Ingestion, OLEM Dir #9200.1-128, Washington, D.C.   July.

EPA. 2017. Work Plan for Investigation of Lead Contamination at the Ethyl Blending and Lead Sweeting Areas at the Wilcox Oil Company Superfund Site, Bristow, Creek County, Oklahoma, October 18, 2017. OSRTI/TIIB, Washington, D.C. October.

EPA. 2017a. Uniform Federal Policy Quality Assurance Project Plan for The Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area at the Wilcox Oil Superfund Site, Bristow, Creek County, Oklahoma Revision 2, October 12, 2017. OSRTI/TIIB, Washington, D.C. October.

Interstate Technology and Regulatory Council (ITRC). 2013. Incremental Sampling Methodology: Representative Sampling, Confident Decisions. A web-based document available at http://www.itrcweb.org/ism-1/

Lockheed Martin ERT (LMS). 2016. Trip Report: November 30 through December 16, 2015, Wilcox Oil Company Superfund Site. Draft. Work Assignment No. ERT-277. 15 April.

United Stated Department of Agriculture (USDA) Natural Resources Conservation Services. Wind Rose Diagram for Tulsa, OK. (www.wcc.nrcs.usda.gov/ftpref/downloads/climate/windrose/)